NASA-CR-54843

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 8
For Quarter Ending July 15, 1965

EDITED BY E. E. HOFFMAN

prepared by
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 8

Covering the Period

April 15, 1965 to July 15, 1965

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-2547

November 23, 1965

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CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Cont'd)

LIST OF TABLES

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from April 15, 1965 to July 15, 1965, of a program to develop a Prototype Corrosion Test Loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in the primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment to the water cooled vacuum chamber. The immediate corrosion test design conditions are shown below; it is expected that the temperature could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr/ft^2

The development program includes the construction and operation of three Cb-1Zr test loops, each of which are being used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers has completed 2650 hours of scheduled testing. This loop was operated at a maximum temperature of 2065°F and a pump inlet temperature of 1985°F. The Prototype Corrosion Test Loop, a two-loop system, which includes a boiler, turbine simulator and condenser in addition to the above components has been fabricated, instrumented, and installed in the test facility and is currently being checked out prior to test start-up. This facility will be used to develop and endurance test (2500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly progress reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures and test results. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specification, purification of potassium and sodium, and inert gas purification and analyses.

II SUMMARY

The fabrication of the Prototype Loop was completed and the assembled loop mounted on the test chamber spool piece was transferred to the test site.

Purification of the alkali metals for the Prototype Loop was performed.

Installation and instrumentation of the Prototype Loop was completed and the loop and spool piece were mounted on the test chamber sump. Following sealing of the test chamber, the system was baked out under vacuum for 180 hours.

The filling, flushing and sampling of the transfer system and the primary and secondary circuits of the loop was performed. Samples of sodium and potassium taken after circulating through the loop for 1 hour at 500°F analyzed less than 10 ppm oxygen.

Checkout tests of the Prototype Loop equipment and controls were conducted and calibration of system instrumentation was completed prior to increasing system temperatures and flows to the design conditions.

The results of the 5000-hour, 2000°F refluxing potassium capsule test to determine the compatibility of Mo-TZM alloy in a Cb-1Zr containter indicate that no significant corrosion problem exists in this system at temperatures equivalent to those planned for the Prototype Loop test.

III PROGRAM STATUS

A. Prototype Corrosion Loop Fabrication

Early in the report period, the final assembly phase of the Prototype Loop fabrication was completed. All the major Cb-1Zr subassemblies were positioned on the support frame and vacuum tank spool section. The loop welding fixture was then positioned inside the support frame and the attachments were made to the loop. This step permitted the loop to be supported in the exact location required for loop operation. The loop support structure was then disassembled and the loop, supported by its welding fixture, was removed from the vacuum chamber spool section as shown in Figure 1. The position of the loop within the welding chamber is shown in Figure 2.

A total of seven welds and local postweld annealing heat treatments were done to complete the Cb-1Zr welding. During welding, the loop welding fixture was rotated to provide the necessary welding positions. The local postweld anneals were done using radiant tungsten coil furnaces, Figure 3, positioned on the individual joints. Temperatures were monitored by two Pt/Pt-10%Rh thermocouples attached to the joint area. A Cb-1Zr control specimen and overlapping 0.002-inch thick Cb-1Zr foil for additional protection from contamination were included in the heated section. Two postweld anneals were done in sequence during each welding chamber evacuation. The vacua maintained during these seven local postweld anneals ranged between 1.3×10^{-5} and 7.4×10^{-6} torr.

Following the annealing of the welds, the Prototype Loop, supported on the welding fixture, was removed from the welding chamber and repositioned in the vacuum tank spool section. The loop is shown in Figure 4 just prior to positioning in the spool section. The loop support structure was then assembled to provide permanent support for the loop. After removal of the welding fixture, the Type 316 stainless steel fill and dump lines, and gas pressure lines were welded to the vacuum chamber ports. Also, stainless steel components of the EM pumps and vacuum feedthroughs for the slack diaphragm transducer were welded in position. Several minor adjustments in the stainless steel support structure were also made to accommodate the position of the potassium flowmeter, heater electrodes, and condenser shield drive assembly. The assembled loop shown in Figure 5 was then transported to the test site for instrumentation.

The stainless steel cans, which contain the reflective foil liner that insulates the Cb-1Zr pump duct from the can walls, were then slipped over the pump ducts and welded to the spool piece flanges. Welding of the two pressure transducer capillary tube feedthrough discs to the spool piece flanges completed the assembly of the loop. These flanges are shown in Figure 5, which is a photograph of the assembled system at the test site prior to initiation of loop instrumentation.

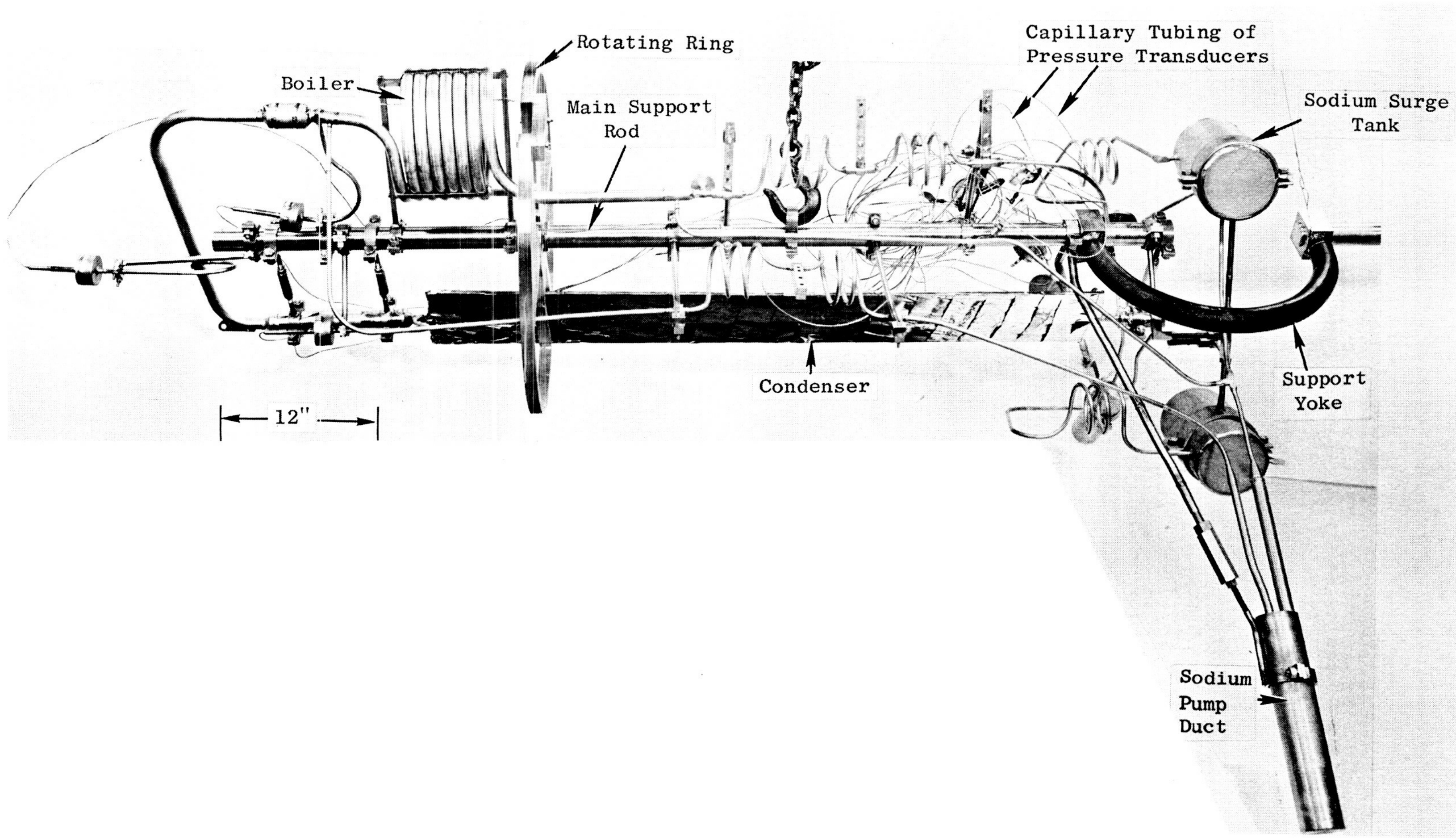


Figure 1. Prototype Corrosion Loop Mounted on Welding Fixture. (Orig. C65051297)

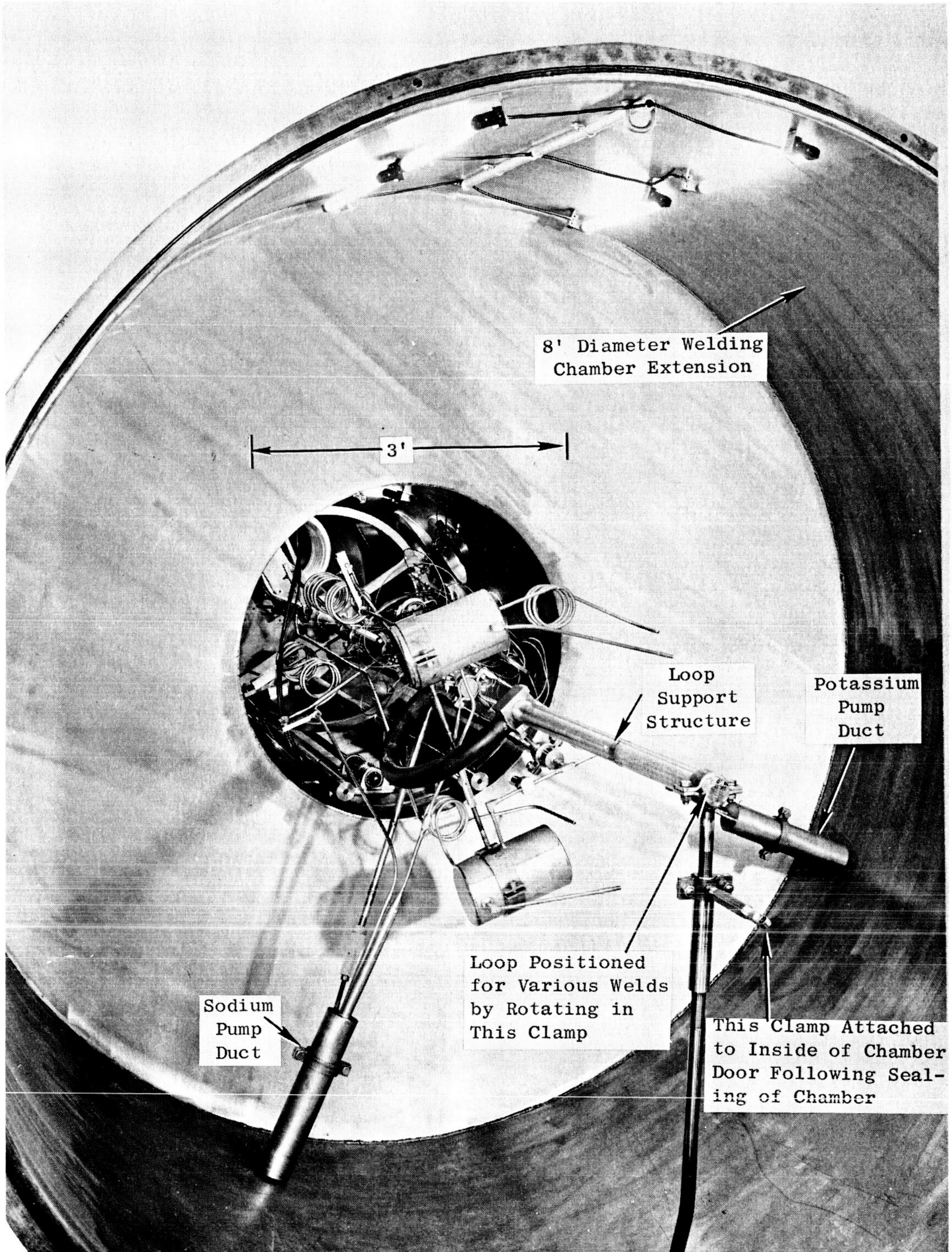
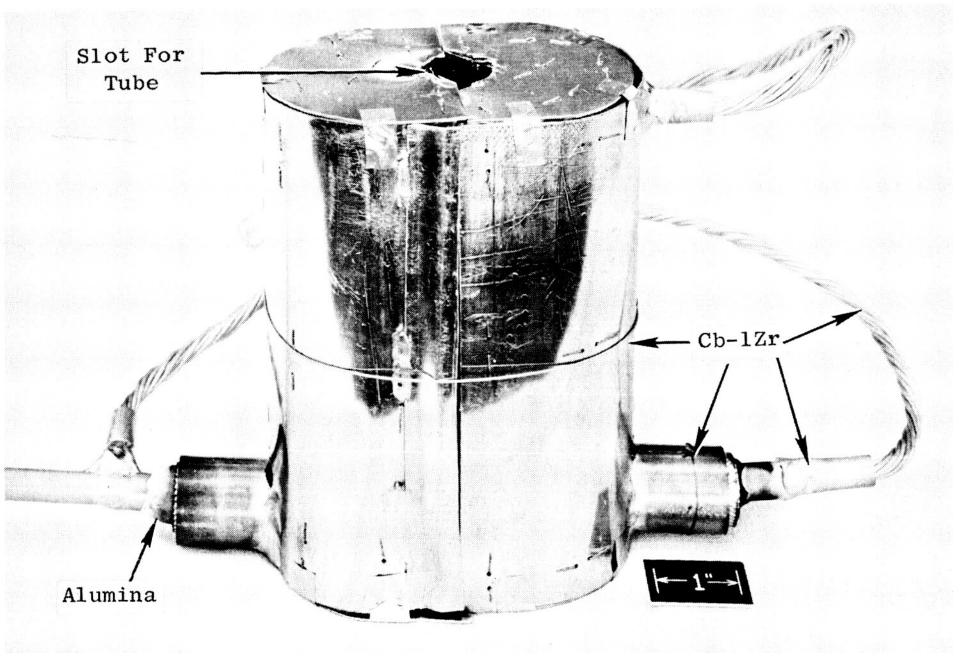
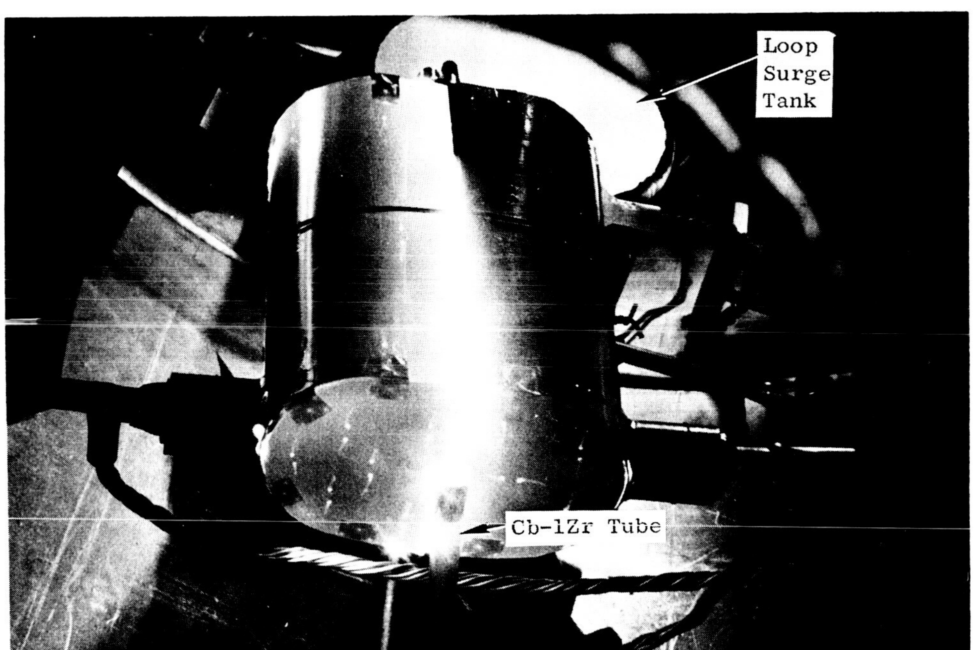


Figure 2. Prototype Corrosion Loop Positioned in the Welding Chamber During Final Assembly. (Orig. C65051295)



(a)



(b)

Figure 3. Refractory Metal Furnace for Vacuum Annealing of Cb-1Zr Loop Weldments in the Welding Chamber. Helical Heater Elements of Tungsten Wire and Eight Layers of Tantalum Reflective Foil Insulation Used to Achieve 2200°F Annealing Temperature. View (a) Shows Furnace Prior to Mounting on Loop Tube Weldments and (b) Shows Furnace in Operation. (a) (Orig. C65032922)
(b) (Orig. C65032301)

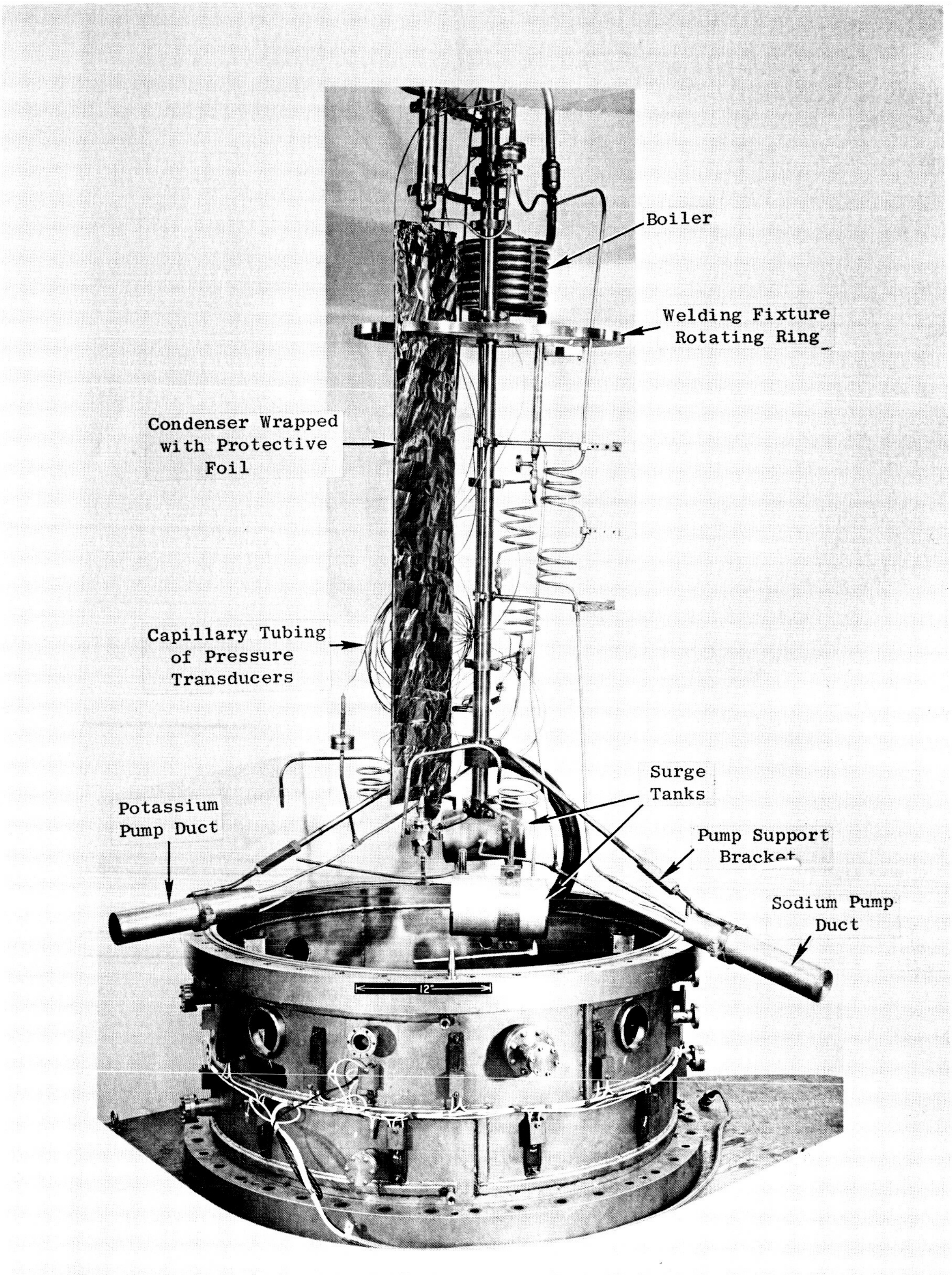


Figure 4. Prototype Corrosion Loop Prior to Installation in the Vacuum Chamber Spool Section. (Orig. C65051298)

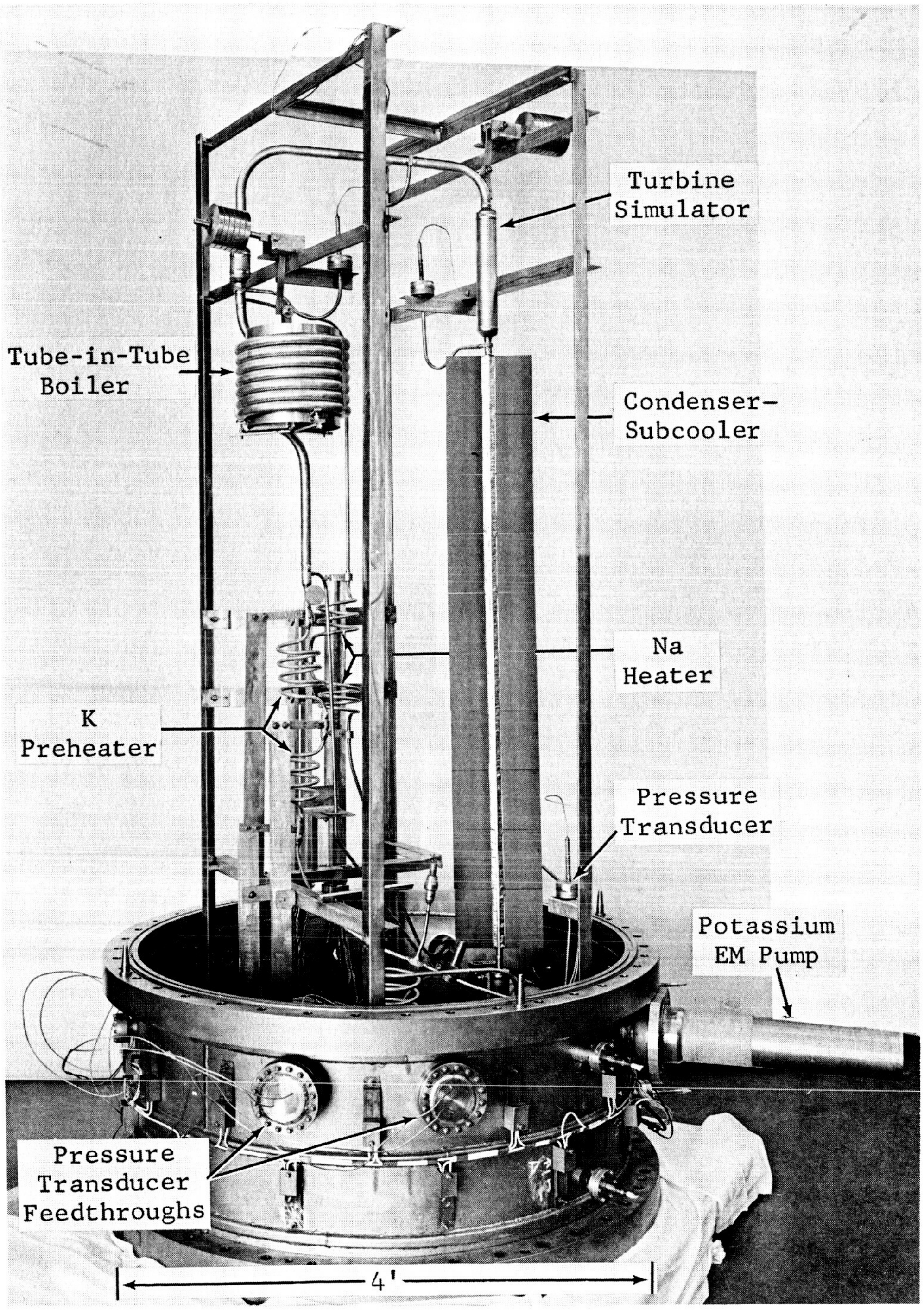


Figure 5. Prototype Corrosion Loop Following Installation in the Test Chamber Spool Piece. (Orig. C65051802)

B. Alkali Metal Purification and Transfer Systems for the Prototype Corrosion Loop

The alkali metal purification and transfer system was described in considerable detail in the previous progress report (1). During this report period the potassium purification system was completed, helium leak checked, outgassed, and used to distill 27.5 pounds of potassium, which had previously been outgassed at 450°F and hot trapped for 50 hours at 1300°F. The ultrahigh vacuum system for the potassium still was helium leak checked and no leaks were found at the detection limit of the instrument which is 5×10^{-11} std. cc of air per sec. This system was outgassed at 300°F until the pressure rise rate was less than one micron-liter per hour while hot. The still itself was also helium leak checked and outgassed. No leaks were found at the detection limit of 5×10^{-11} std. cc of air per sec. During the outgassing, the still pot temperature was 800°F, the condenser temperature was 400°F, and the receiver temperature was 320°F. The ultimate pressure with the still hot was 1.7×10^{-7} torr. With the system at room temperature, the pressure was 3.9×10^{-8} torr, and the pressure rise rate was 0.4 micron-liter per hour.

The potassium was distilled at 500° to 600°F at an average rate of about one pound per hour. The coaxial tube condenser (Cb-1Zr tube inside of a stainless steel tube) with potassium in the annular space operated quite well. A flow of only 10 SCFH of plant air through the copper cooling coils was required to maintain the condenser temperature in the 250° to 350°F range. The distillate was sampled and analyzed for oxygen and metallic impurities. The analytical results are shown in Table I.

The purification of the sodium for the Prototype Loop and the results obtained were reported in a previous report (2). These analytical results are also included in Table I.

Additional information regarding the filling, flushing and sampling of the sodium and potassium circuits of the Prototype Loop is presented in Section D of this report.

C. Installation and Instrumentation of the Prototype Corrosion Loop

Following completion of loop fabrication and mounting of the loop on the test chamber spool piece, the loop was transferred from the fabrication area to the test operation area by pneumatic-wheeled truck traveling at a maximum speed of 5 miles per hour. During the transfer, the entire loop was covered with a polyethylene bag and all open gas pressurization and liquid metal fill lines were covered with polyethylene sheet held in place with plastic tape.

The test loop was positioned near the vacuum system, and the air shelter which was shown in an earlier report (3) was assembled over the test loop. The air shelter, which was used previously while instrumenting Loop II, is a semi-spherical reinforced polyethylene bag approximately 15 feet in diameter and 15 feet in height and is supported by 0.2 psi air pressure supplied by a small centrifugal blower. The initial placement of the loop was at ground level rather

TABLE I. ANALYTICAL RESULTS FOR LIQUID METALS USED IN THE PROTOTYPE CORROSION LOOP

IMPURITY CONTENT

REPORTED AS PPM OXYGEN IN K OR Na AND PPM METALLIC IMPURITY IN KCl OR NaCl

Impurity	As Received Na	As Received K	After Hot Trapping Na	After Hot Trapping K	After Distilling K	After Flushing Transfer System Na	After Flushing Transfer System K	After Circulating Through Loop for 1 Hr. at 500°F Na	After Circulating Through Loop for 1 Hr. at 500°F K
Oxygen	12	4,6	4,9	4,6	4,4	12,17	6,7	5,7	6,9
Ag	<1	<1	<1		<1			1	<1
Al	15	1	<1		<1			<1	<1
Ca	10	5	10		1			1	1
Cb	--	<1	<1		<1			<1	<1
Co	<5	<1	<1		<1			<1	<1
Cr	2	<1	<1		<1			<1	<1
Cu	2	<1	<1		<1			<1	<1
Fe	4	1	<1		<1			<1	<1
Mg	1	<1	<1		<1			<1	<1
Mn	<1	<1	<1		<1			<1	<1
Mo	<5	<1	<1		<1			<1	-
Na	--	5-25	-		25			-	25
Ni	<1	<1	<1		<1			<1	<1
Pb	<5	<1	<1		<1			<1	<5
Si	<10	5	<1		<1			<5	<5
Sn	<5	-	<5		<1			<5	<5
Ti	<5	<1	<1		<1			<1	<1
V	<1	<1	-		-			<10	<5
Zr	<10	<5	<5		<5			<5	<5

than in the final test position on the vacuum system sump which allowed greater accessibility during instrumentation. All personnel were required to wear clean, white dacron gloves, shoe covers, and coveralls while working in the air shelter.

Following erection of the shelter, the temporary support brackers used in the fabrication and transfer of the loop were removed and permanent brackets were installed along with instrumentation channels for routing thermocouple and pressure sensor lead wires. The OFHC copper bus bars for both the potassium preheater and sodium heater were installed at this time, since they also serve as structural support members to maintain the helical heater coils in position.

The locations and designations of the 79 thermocouples which were installed on the loop were indicated in an earlier report (4). Sixty-six of the loop thermocouples are of the wall junction or surface type with each thermocouple wire spot welded directly to the loop component and held in position by the techniques illustrated in Figures 6 and 7. Thirteen of the thermocouples were of the well type with the thermocouple wires joined together prior to insertion into a well. The well type thermocouples were specified for components where a reliable but average temperature was required and the geometry of the component permitted the addition of a well or a drilled hole. These included the inlet and outlet to the EM pumps, the turbine simulators, and the potassium preheater. Thermocouples were also placed in three wells of varying depth in the condenser fin near the top of the condenser.

The procedure used in installing the loop thermocouples was a departure from the method used in Loops I and II (5). For these earlier tests the tungsten-rhenium thermocouple wire was routed from the loop through the thermocouple feedthrough to a constant temperature reference junction located outside of the vacuum tank. Copper lead wires were then used between the reference junction and the recorder. In both Loop I and Loop II problems were encountered with the thermocouple vacuum feedthrough because of the difficulty in brazing tungsten-rhenium wire to the nickel tubulation of the vacuum feedthrough. Not only was the assembly difficult to seal effectively, but a number of failures occurred on the W-3%Re wire near the brazed joint during installation and later during loop operation. To minimize these problems, the reference junction was placed inside of the vacuum chamber and copper lead wire was used from the junction block through the vacuum feedthrough to the recorder. This method is illustrated in Figure 8. The assembly shown in Figure 8(b) consists of four octal (8-tube) feedthroughs* welded into a 6-inch flange.

The reference junction block as shown in Figure 9 consists of eight 99.7% Al_2O_3 tube terminal strips mounted on a 1/4-inch thick copper plate which is bolted to the wall of the sump of the chamber. The entire assembly was shielded from the hot portions of the loop by means of a stainless steel optical baffle to minimize temperature gradients in the junction block and to maintain a lower absolute temperature approaching that of the water cooled vacuum chamber wall. The temperature of each of the alumina tube thermocouple reference junction blocks, which is required to correct the temperature readings of the loop, is measured by copper/constantan thermocouples located on one junction terminal of each of the eight alumina tubes.

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*Varian Associates, Vacuum Products Division, Model 954-5015

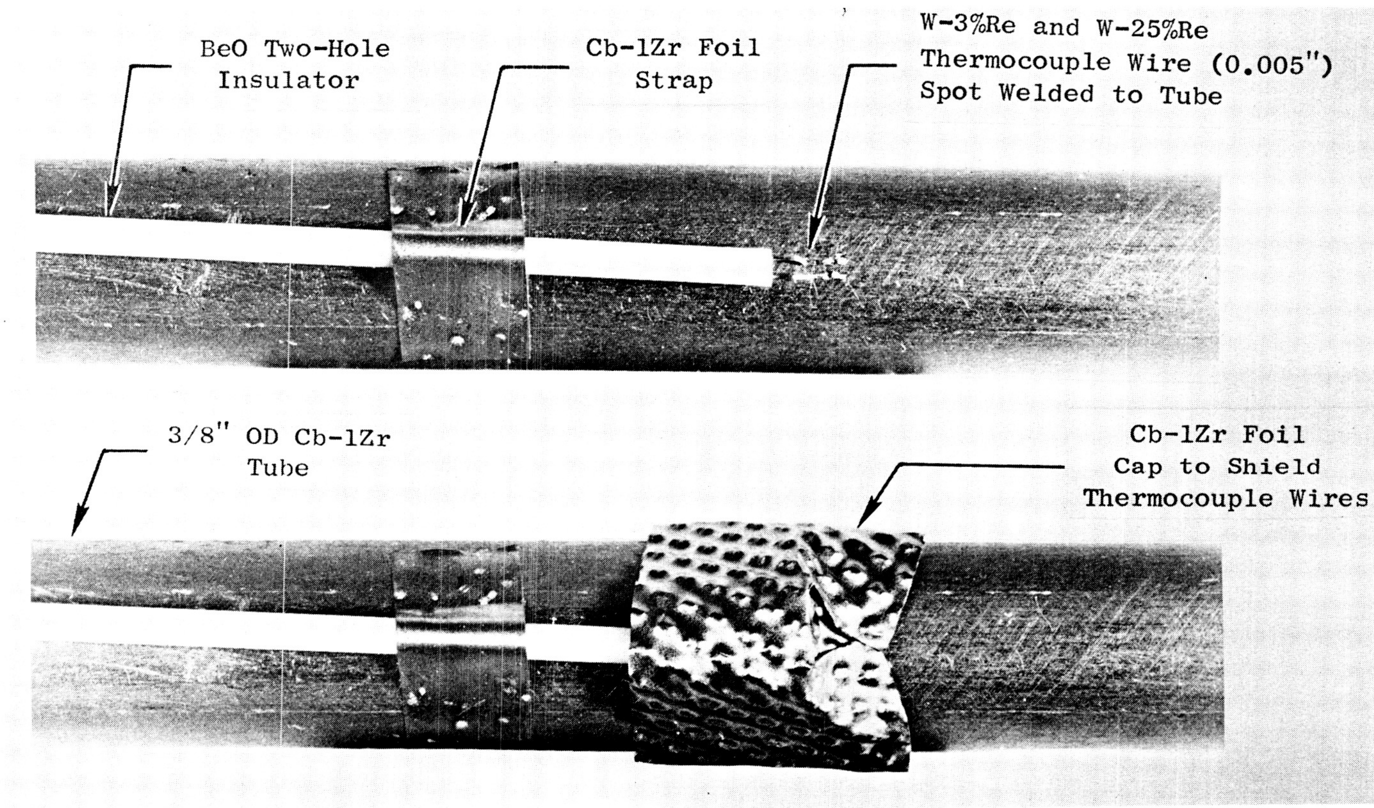

Figure 6. Method Used to Mount W-3%Re/W-25%Re Thermocouples Wires on Cb-1Zr Alloy Tubing for the Prototype Corrosion Loop Test. (C65062199)

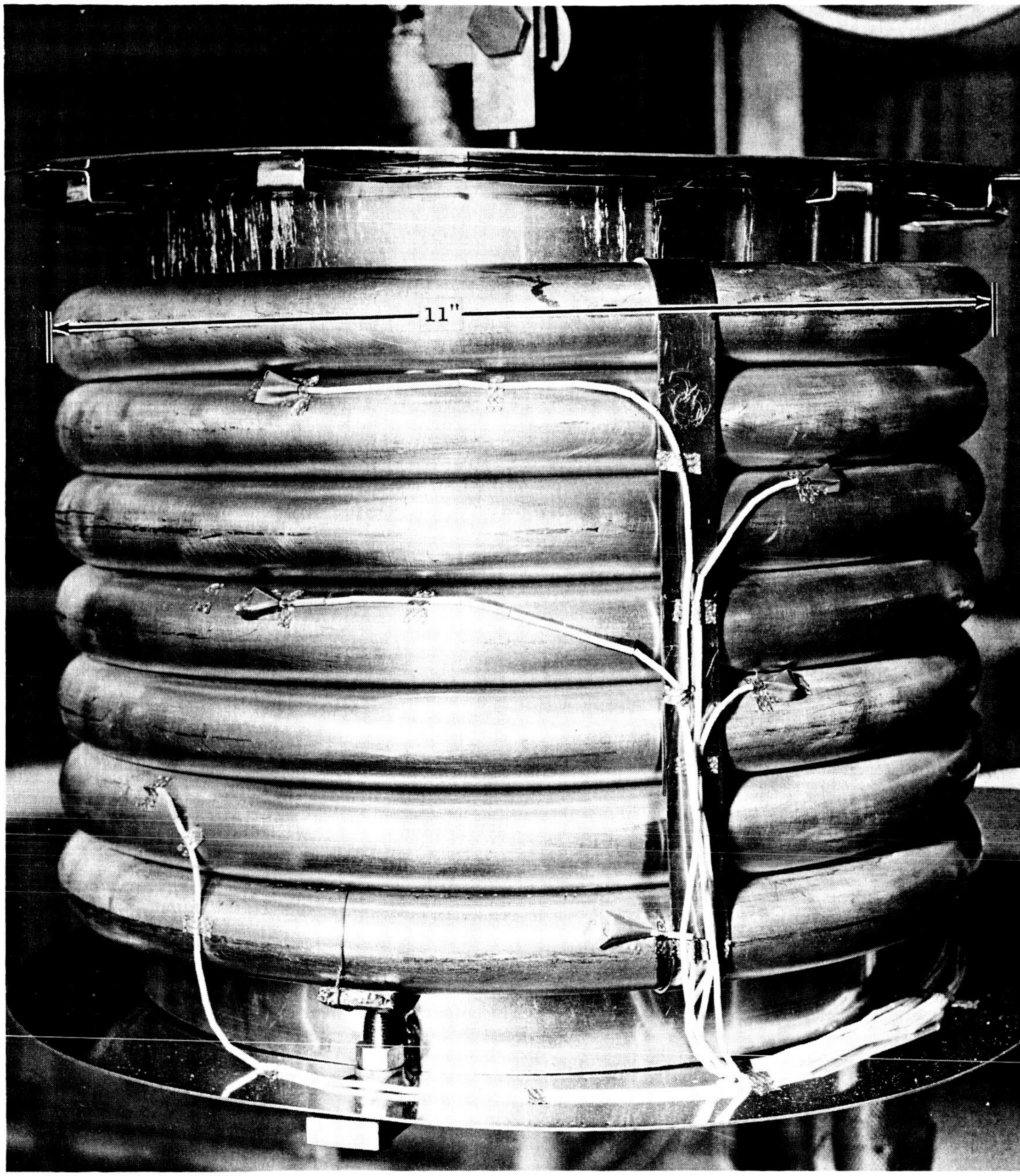

Figure 7. Photograph of Tube-in-Tube Helical Boiler of the Prototype Corrosion Loop Showing the Method Used to Mount Boiler Thermocouples. (Orig. C65061613)

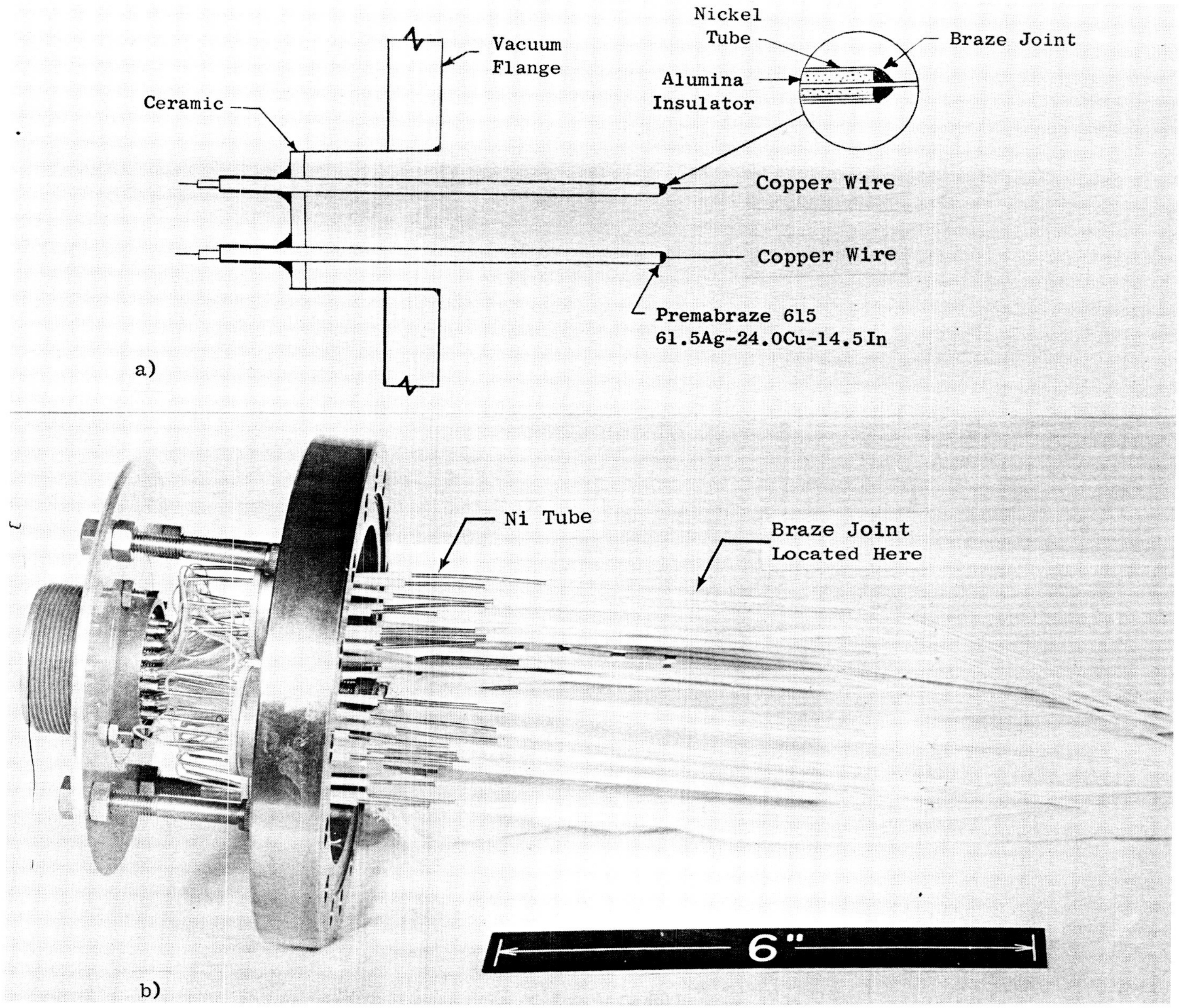

Figure 8. Typical Thermocouple Lead Wire Feedthrough Flange.
a) Sketch Illustrating Method Used to Seal Wire to Nickel Tube.
b) Feedthrough Containing 32 Lead Wires. (C650621103)

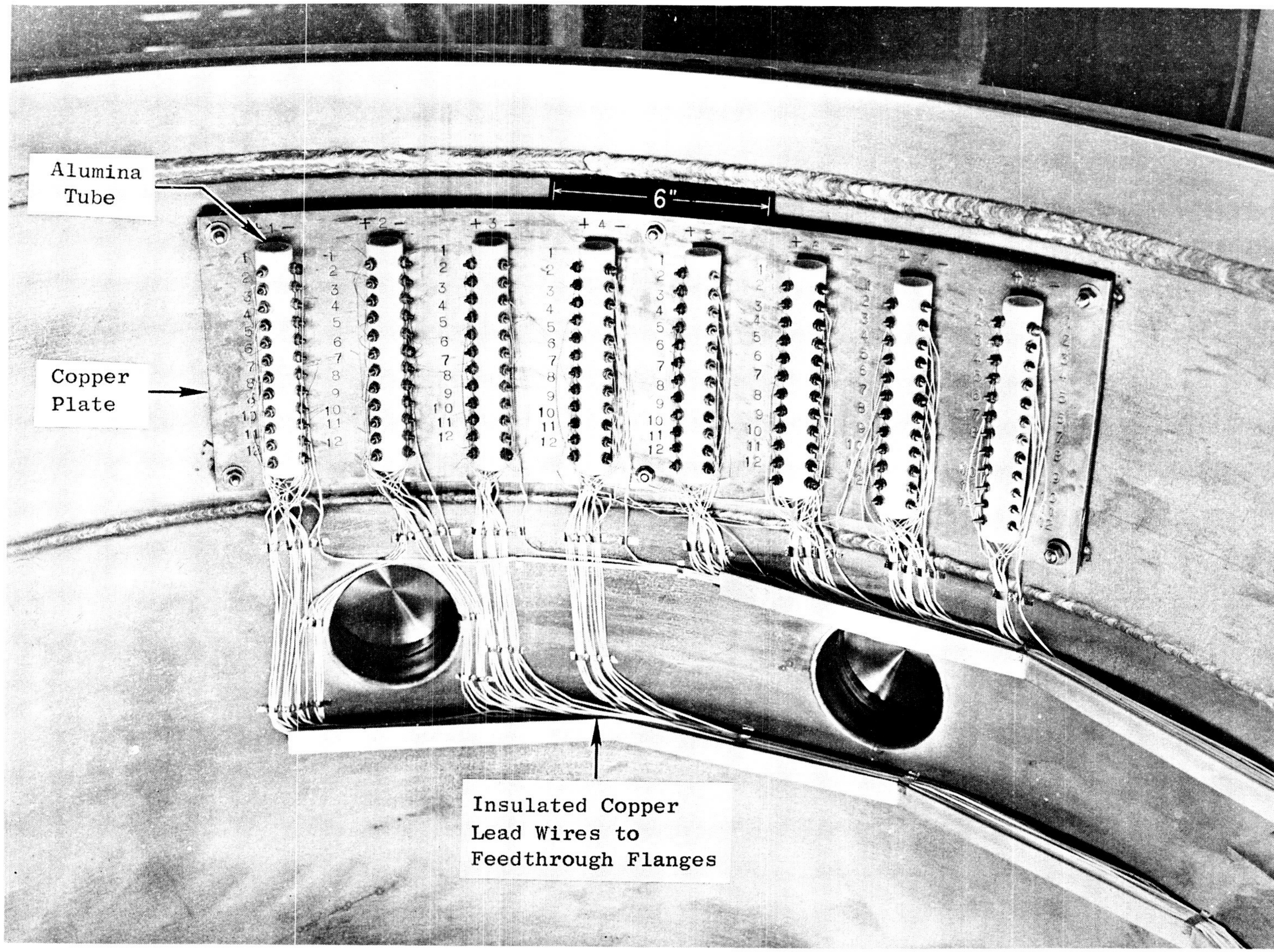

Figure 9. Thermocouple Wire Terminals (192 Total) Located in the Sump of the Test Chamber for the Prototype Corrosion Loop. (C65062196)

An additional advantage of locating the reference junction block inside of the vacuum chamber is that the routing of the thermocouple lead wires from the terminal strip to the recorder can be completed prior to mounting of the loop and spool piece on the chamber flange and can save considerable time in the installation schedule.. The method used to mount the thermocouple lead wire feedthroughs in the 6-inch OD crosses is shown in Figure 10.

Thermal insulation consisting of multiple layers of Cb-1Zr foil was simultaneously applied to the loop as the thermocouples were installed. The foil used on all circular pipe sections was 0.002-inch thick x 0.5-inch wide which had been dimpled by passing the foil between a hardened steel, coarse knurled roller working against a hard plastic sheet. The dimpling process used in preparing the insulation is illustrated in Figure 11. The effective thickness of the foil after dimpling was between 0.009 to 0.012 inch. The insulation was attached to the tube by spot welding the foil to the tube and to itself as succeeding layers were applied. A minimum number of spot welds were used to minimize conduction heat losses through the foil. A molybdenum electrode was substituted for the copper spot welder electrode to avoid contamination of the foil surfaces with copper and an argon cover gas was used to protect all welded areas from oxidation.

The type of insulation used on the various loop components is shown in Figure 12. The insulation for the boiler was pre-assembled and installed around the boiler after the thermocouples were attached to the boiler tube. The assembly consisted of a center cylinder, two outer semi-cylindrical halves, and a top and bottom end shield.

After completion of the instrumentation and insulation of the loop, the loop was covered with a polyethylene bag and the clean room was dismantled. The loop and spool section on its plywood pallet were lifted by a fork truck above the vacuum sump, and the bell jar was lowered and then bolted to the spool flange. The bell jar and the attached spool section were raised together off the pallet and then lowered to the sump. The flanges were bolted together and the vacuum chamber was evacuated and helium leak checked. This sequence was required to ensure a leak tight joint between the sump and lower spool flange because the spool becomes fixed in position when the EM pump windings for the two pumps are attached to their spool flanges and the gas pressurization and liquid metal fill lines are welded to their spool piece ports.

Upon completion of the helium leak check, the vacuum system air release valve was opened and the chamber was returned to atmospheric pressure. The bell jar was hoisted into the penthouse and the final instrumentation and the installation of loop components, which could only be completed with the loop in its final test position, was resumed.

The metering and on-off isolation valves were installed with their actuating system which consists of an ultrahigh vacuum rotary feedthrough with a torque rating of 6 ft-lbs* connected by a 0.31-inch diameter flexible stainless steel

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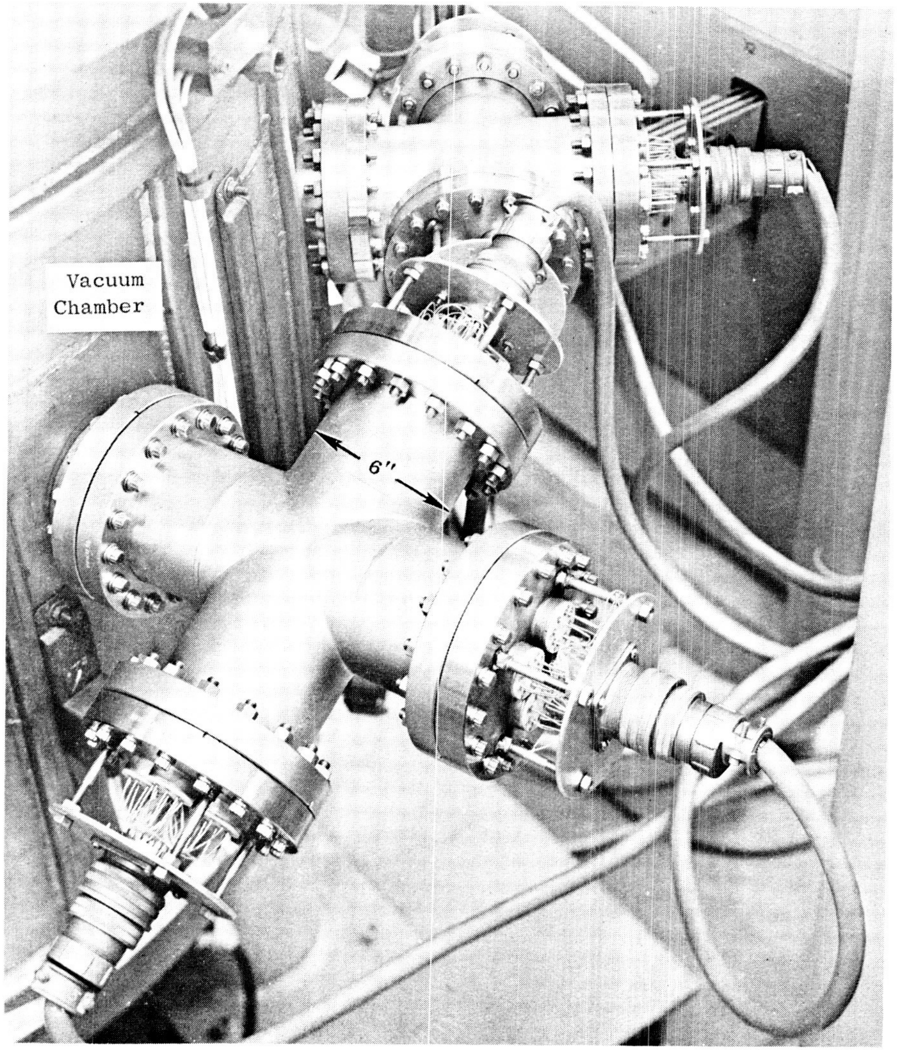
* Varian Associates, Model No. 954-5039.



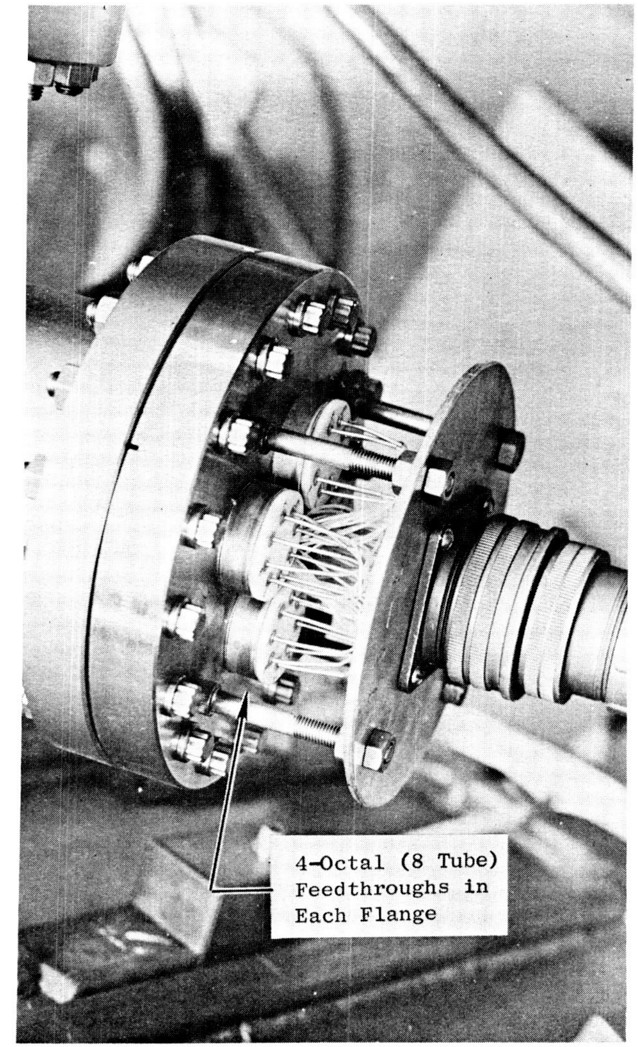


Figure 10. Six Thermocouple Lead Wire Feedthrough Flanges with 32 Wires in Each Flange. An Enlarged View of One of the Flanges is Shown on the Right. (C65062189)

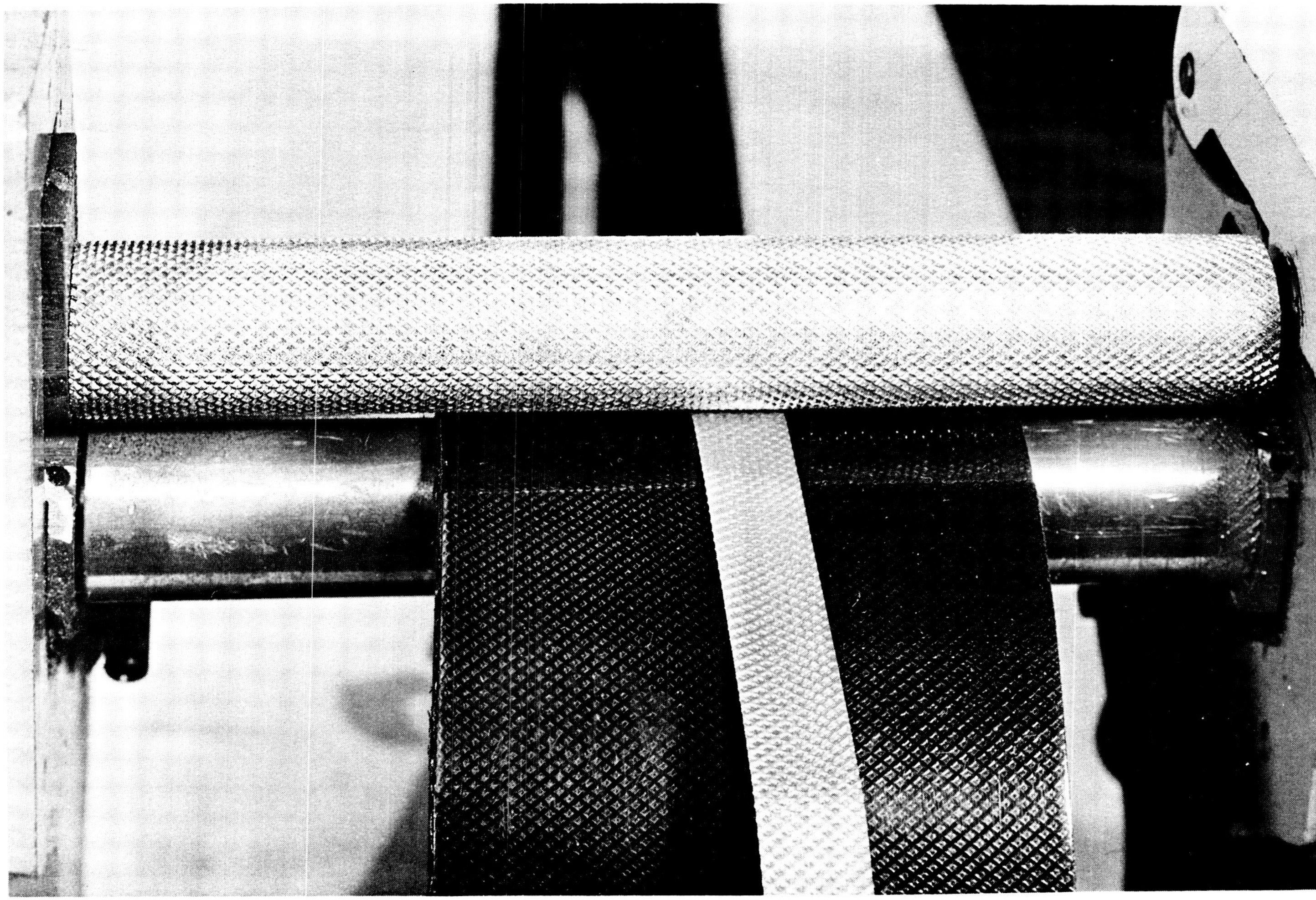


Figure 11. Dimpling of 0.5-Inch Wide x 0.002-Inch Thick Cb-1Zr Alloy Foil Used to Insulate Loop Components. (C64102722)

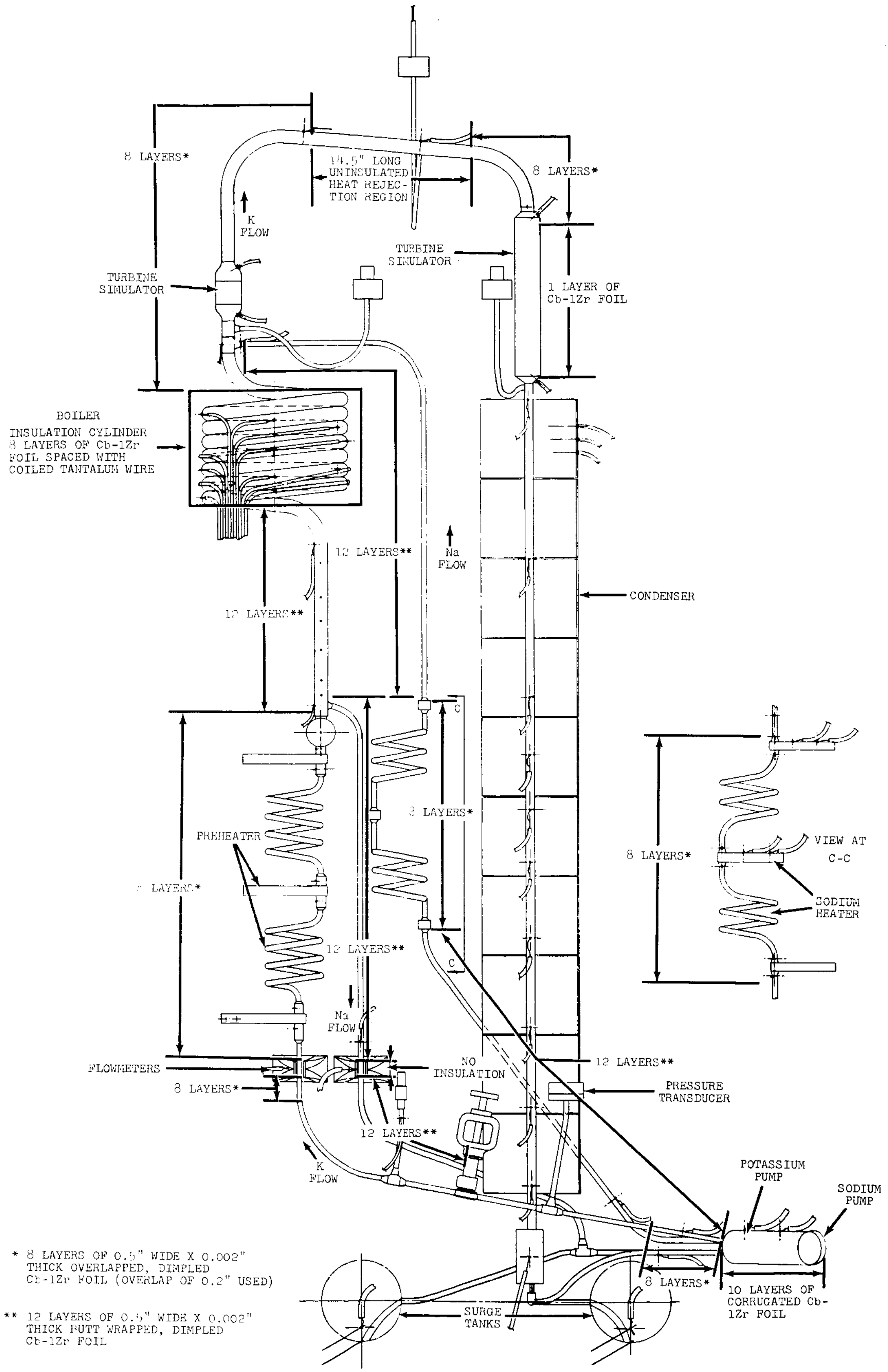


Figure 12. Prototype Corrosion Loop Showing the Types of Reflective Foil Thermal Insulation Used on the Various Loop Components.

cable to a 5:1 right angle gear drive mounted on the valve yoke. The right angle gear drive is required to eliminate a compound bend in the flexible cable because the rotary feedthroughs are located below the valves. Two views of the metering valve and the valve drive assembly are shown in Figure 13. The enlarged view in Figure 13b shows the pinion drive gear and the spur gear which is mounted on the valve stem. By sighting on the wire shown and the 72 divisions laid out on the face of the gear, with the aid of a telescope located at one of the chamber view ports, precise measurements of valve adjustments will be obtained during loop operation. The flexible cables shown were cleaned ultrasonically and baked out at 500°F under a vacuum of less than 1×10^{-3} torr for 100 hours before installation. No lubrication of any type was used in the assembly. The various parts of the valve itself were described in the previous progress report (6).

The adjustable condenser shield assembly was the last component to be installed. A portion of the shield including the rack and spur gear drive system and four of the eight movable shield fins constructed of Cb-1Zr are shown in Figure 13a.

A final check on the loop instrumentation was then made and the stainless steel mirrors which were attached to the support structure were adjusted to give a full view of all components which were not directly visible through the view ports. The loop and auxiliary components are shown in Figure 14 following completion of the installation task.

The bell jar was then lowered and bolted to the spool piece flange. The vacuum system bakeout heaters were turned on and the chamber was evacuated to 10 microns and then backfilled with dry nitrogen gas. The system was again rough pumped with the liquid nitrogen chilled sorption pumps. The sorption pumping system was then valved off, and the getter-ion pumps were turned on when the pressure at the roughing station was reduced to less than 5 microns.

Varian Associates, the manufacturer of the test chamber and pumping system, recommended that the titanium sublimation pumps be turned on when the chamber pressure was reduced to 15 microns by the sorption pumps. This procedure reduces the time required to lower the chamber pressure sufficiently to turn on the getter-ion pumps and confine the glow discharge. The "glow discharge" period is associated with high pressure conditions (5-20 microns) when the getter-ion pump voltage is low (approximately 200 volts), the current is high, and the pumping rate is low. In order to save the titanium sublimation pumps for use during the period of loop startup when high gas loads are anticipated, the sublimation pumps were not used during the initial startup of the getter-ion pumps. The "glow-discharge" pumping condition persisted for three hours before the getter-ion pumps confined the discharge and the high voltage, low current, high pumping rate condition was obtained.

After 180 hours of getter-ion pumping, the total pressure of the system was 5×10^{-6} torr with the chamber on bakeout at a maximum temperature of 500°F. Cooling the chamber to 250°F reduced the system pressure to 4.8×10^{-8} torr. During the bakeout period numerous leak checks were made and several leaks were detected in the main flange and one leak was located in a gas

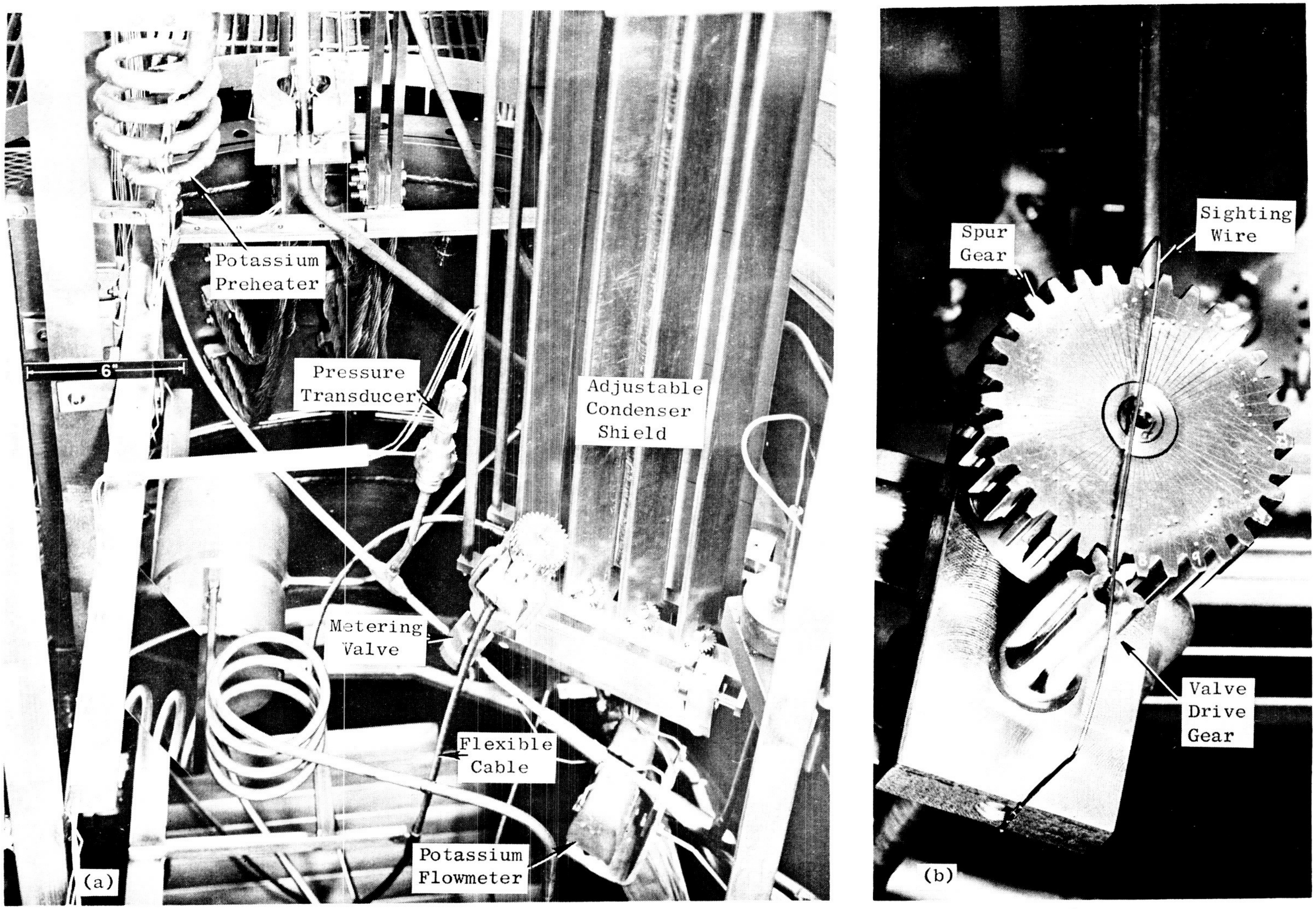


Figure 13. Lower Section (a) of Prototype Corrosion Loop and Enlarged View (b) of the Metering Valve Drive and Sighting Wire Used to Monitor Valve Adjustments.
(a) (Orig. C65063018)
(b) (Orig. C65063016)

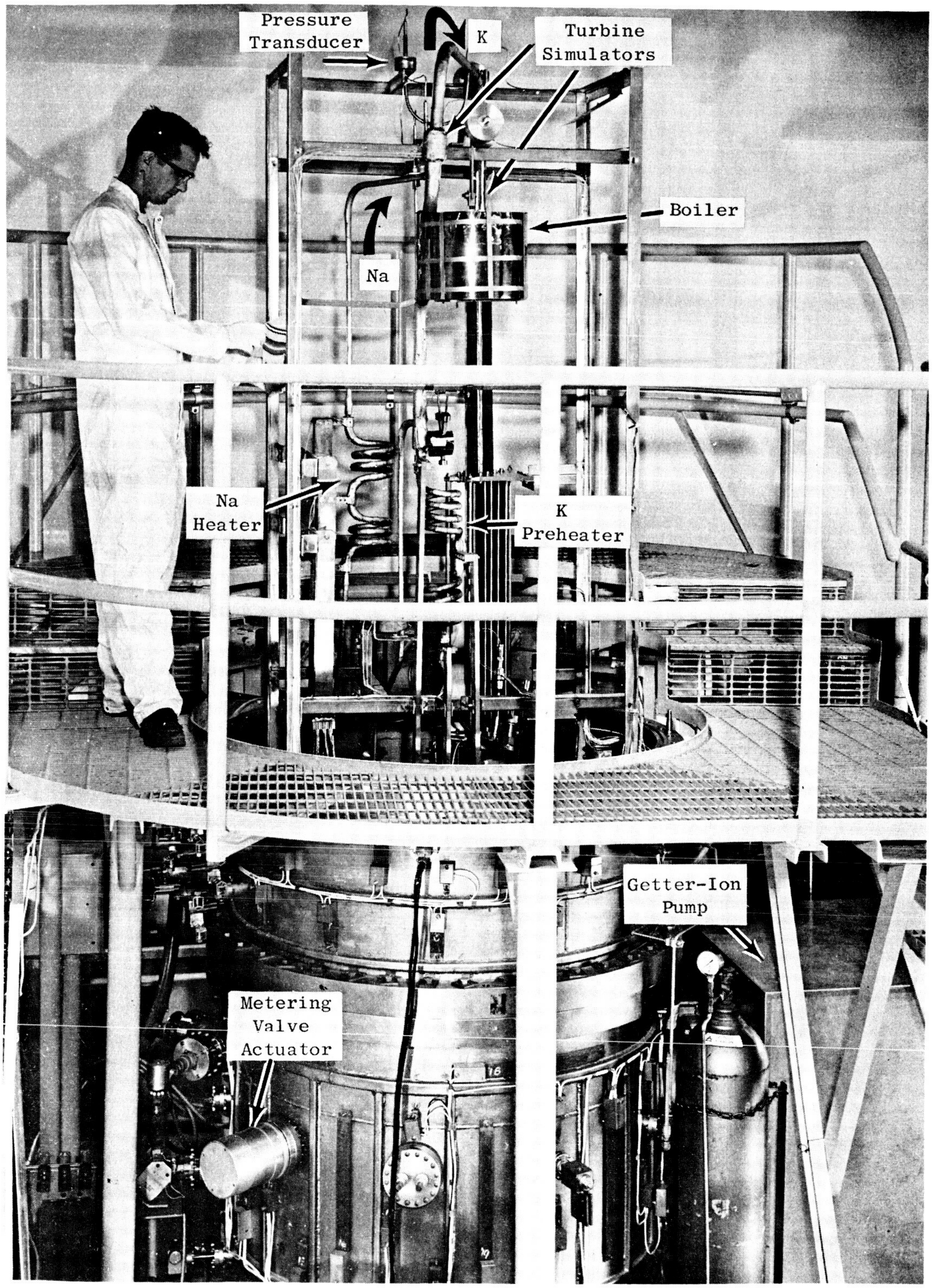


Figure 14. Prototype Corrosion Loop and Test Chamber Prior to Lowering Chamber Bell Jar Onto Spool Piece. (Orig. C65083015)

pressurization feedthrough. The gas pressurization line leak was sealed by welding and the flange leaks were sealed by retorquing all bolts to 225 ft-lbs. Additional leaks in the main flange were encountered and these were attributed to the thermal cycling of the on-off type of controller used in the bakeout system. A desirable feature of vacuum chambers for applications of this type would be a bakeout heater with a proportional controller which would eliminate the thermal cycling and permit a controlled heating and cooling rate for the main flanges. After considerable difficulty, all leaks in the main flange were eliminated and preparations were made to fill, flush, and sample the alkali metal circuits. Chamber pressures during bakeout and test start-up will be included in the next progress report.

D. Filling, Flushing, and Sampling of Alkali Metals in the Prototype Loop System

The sodium and potassium purification systems and the transfer systems were interconnected and attached to the loops. A plan view of this arrangement was shown in a previous report (2). All liquid metal transfer lines outside of the ovens were traced and insulated for heating. Thermocouples were attached to all valves and at numerous other locations in the system to monitor temperatures during the outgassing, liquid metal flushing, and loop filling operations.

The transfer systems and loops were outgassed at temperatures ranging from 250° to 500°F. The pressure rise rates for the combined loop and transfer systems were 0.3 micron-liters per minute for the primary (sodium) circuit and 0.9 micron-liters per minute for the secondary (potassium) circuit while hot. Both systems were helium leak checked hot and no leaks were found at a sensitivity of 5×10^{-11} std. cc of air per sec.

The transfer systems were flushed with liquid metal at 200° to 300°F, and specimens of the liquid metal used for flushing were obtained and analyzed for oxygen. The analytical results are indicated in Table I.

The loops were filled at about 300°F and additional specimens of the alkali metals were obtained after circulating in the loop for one hour at 500°F. The analytical results are also shown in Table I.

E. Checkout of the Prototype Loop Equipment and Controls and Calibration of the Loop Instrumentation

Following the filling of the loop circuits with the operation charges of sodium and potassium, checkout of the loop equipment and controls was continued. The only major problem encountered in the equipment checkout was in the failure of the electrical power supply for the potassium pump. A capacitor used to improve the power factor of the highly inductive load of the pump was defective and caused overheating and arcing in the motor-driven autotransformer used to control the electrical power to the pump. Replacement parts from similar equipment in the laboratory which were not in use were available and the checkout was resumed without serious delay.

Calibration of all pressure sensors which had been originally calibrated before installation with inert gas was repeated with the loop at 500°F and the chamber on bakeout using a calibrated Wallace and Tierman Model No. FS145 pressure gauge as the standard. Corrections for the liquid potassium heads were made for all pressure sensors according to their evaluation above the surge tank. All pressure sensors showed good linearity and excellent repeatability.

The calibration results for slack diaphragm pressure transducer #1 (potassium pump outlet) is shown in Figure 15 and is typical of the results obtained for the otherthree slack diaphragm transducers.

The calibration of the fast response stressed diaphragm with potassium prior to test start-up is shown in Figure 16. This transducer showed a 20% increase in millivolt output in the 0-150 psia range compared to the pre-installation room temperature calibration with argon gas. The temperature of the transducer during the liquid metal calibration was 460°F which would indicate that the transducer is temperature sensitive and should be thermally aged before calibration.

Thermal treatment prior to calibration might reduce the temperature sensitivity observed in this transducer and this treatment will be performed in future tests of this type.

The primary loop thermocouples were checked by operating the sodium circuit at near isothermal conditions and comparing the indicated temperature of each thermocouple with those adjacent to it. A near isothermal condition in the primary circuit was achieved by operating at a high sodium flow rate (2.5 gpm) with the potassium loop empty. The calibration is also useful in computing heat balances in subsequent loop operations because the temperature drop of the sodium during the calibration run can be equated to the heat loss of boiler. The temperature distribution as a function of boiler length at the 1630°F calibration run is shown in Figure 17.

During this calibration os primary circuit thermocouples, the chamber pressure rose from 7.6×10^{-7} torr when the nominal temperature of the sodium circuit was 1200°F to 1.2×10^{-6} torr with a sodium circuit temperature of 1630°F due to outgassing of the system. Although thermocouple calibration runs at temperatures in the range of loop design conditions (2000°-2150°F) were desired, these calibrations will be deferred to the end of the test following dumping of the potassium from the secondary circuit.

The primary and secondary flowmeters were calibrated by comparing the indicated flow based on the theoretical equation (7) with the flow rate obtained by an energy balance across the preheater and heater during all liquid operation. The results of these tests are shown in Figures 18 and 19. The indicated primary flow rate is 37% lower than the flow rate calculated from the energy balance; the secondary flow rate is 32% lower. This discrepancy in flow rates has also been observed in heat transfer tests (8) where the indicated flow rate was as much as 33% lower. The discrepancy between the actual

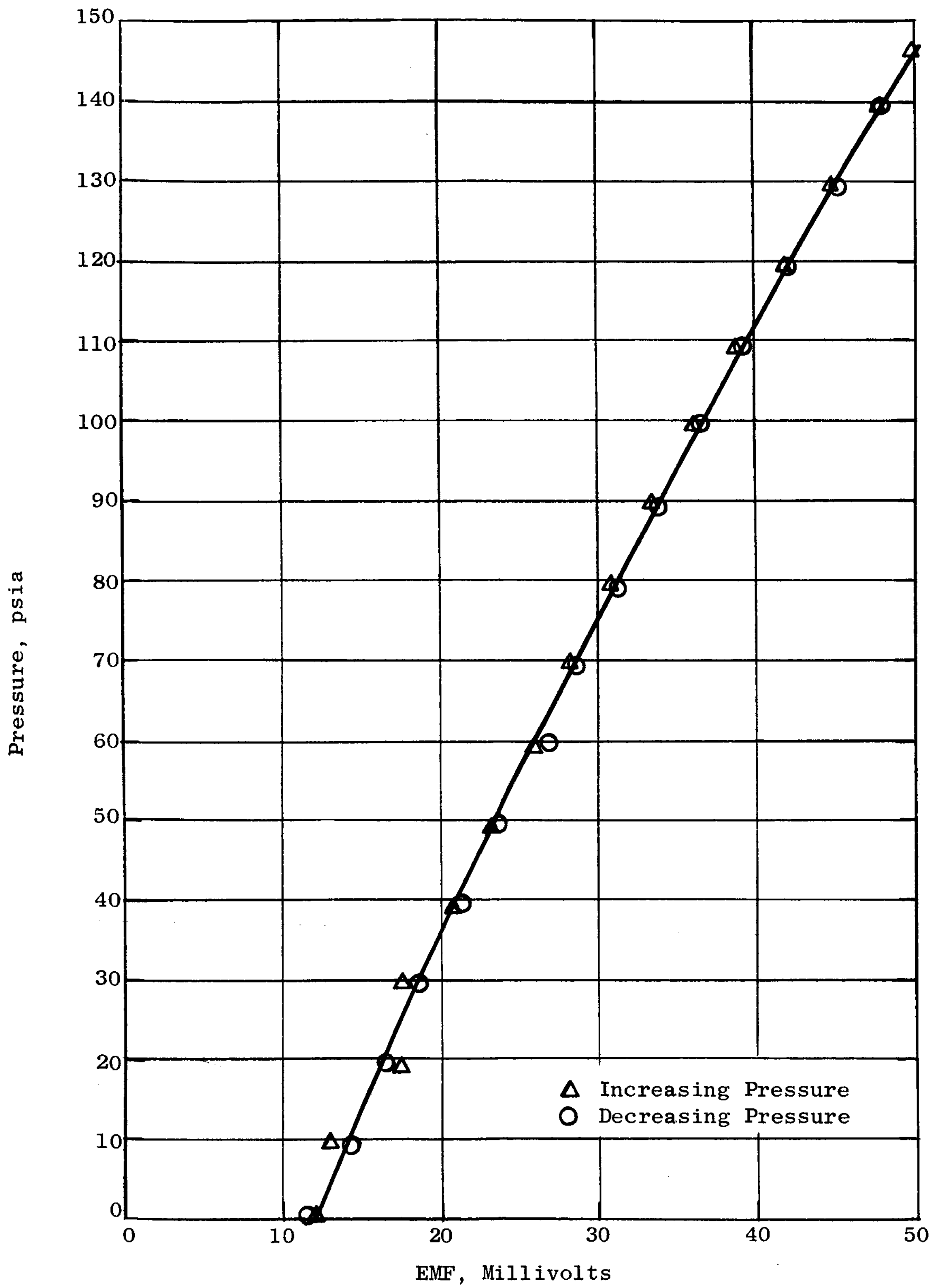


Figure 15. Calibration of Slack Diaphragm Pressure Transducer No. 1 for the Prototype Corrosion Loop.

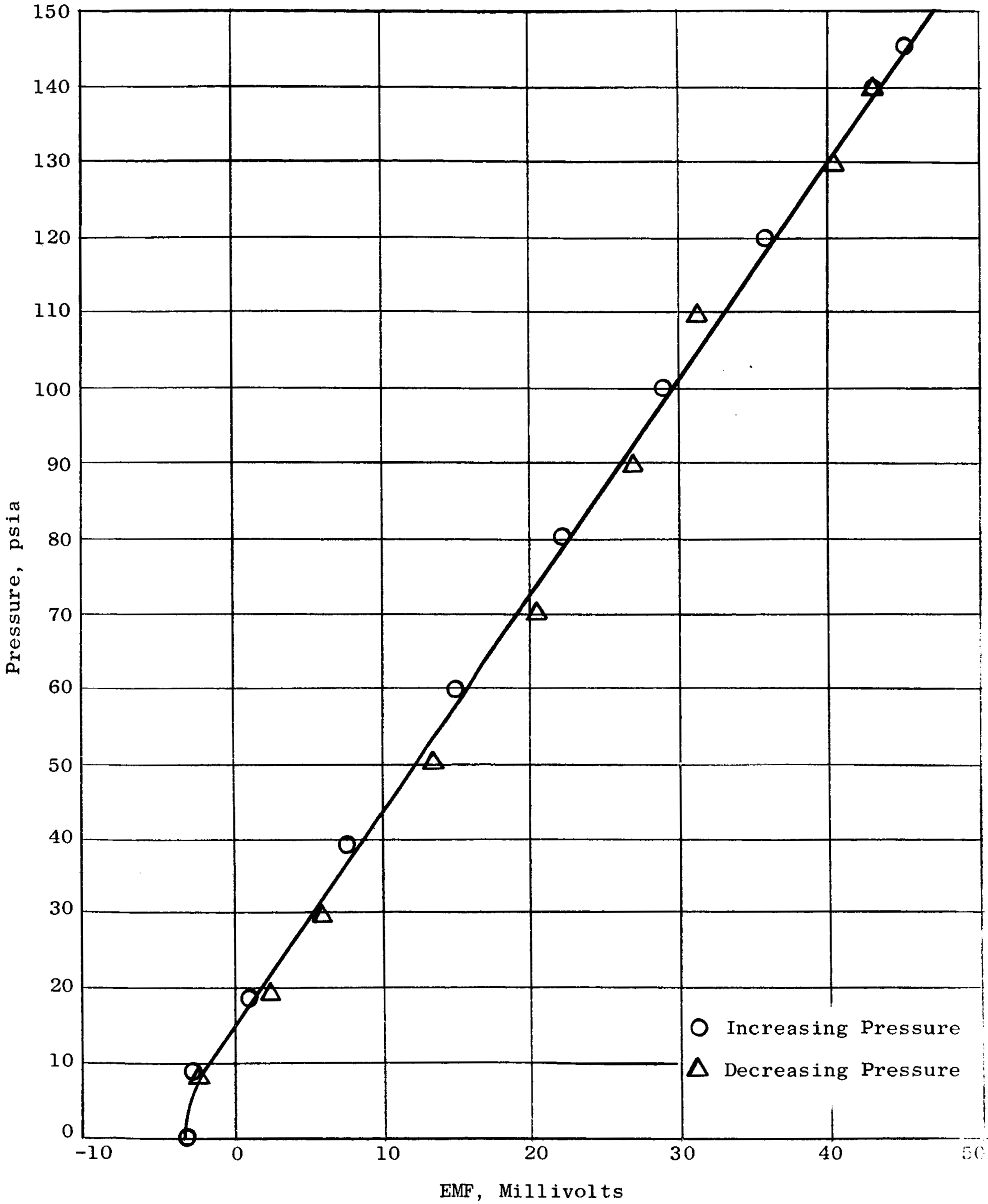
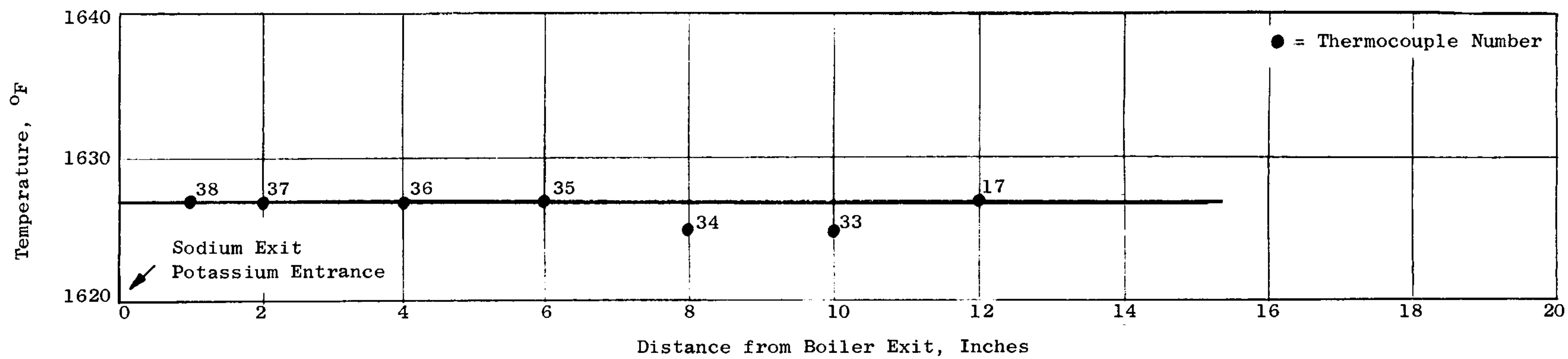


Figure 16. Calibration of Fast Response Stressed Diaphragm Pressure Transducer for the Prototype Corrosion Loop.



Expanded Scale Showing Thermocouple Calibration Points in Plug Section of Boiler.

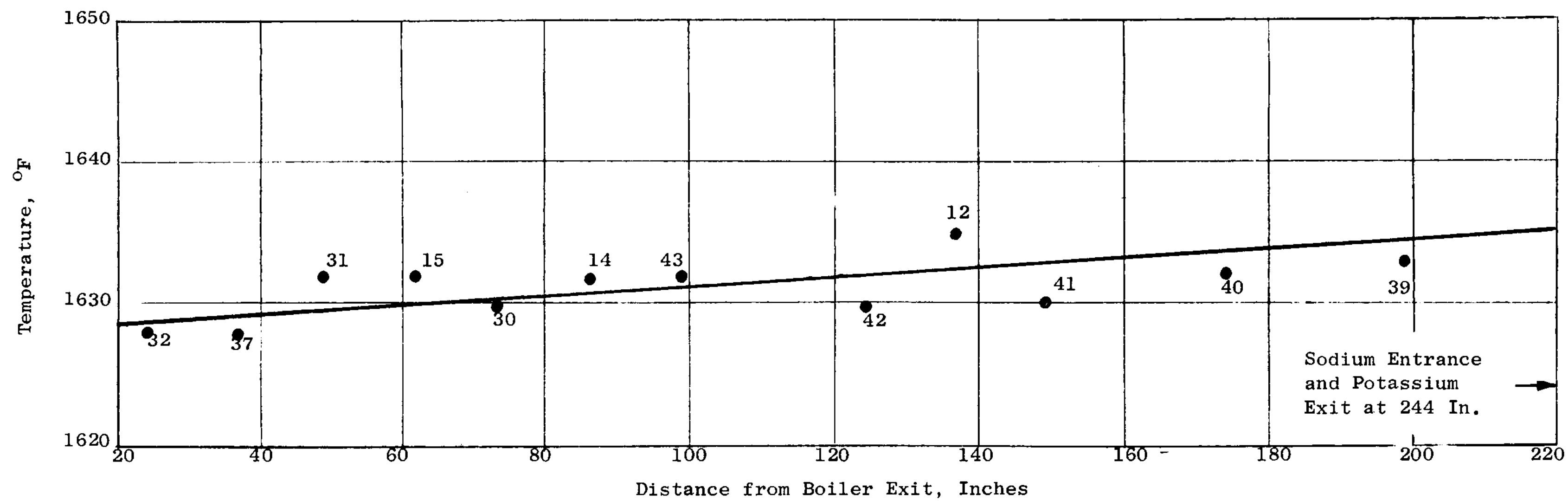


Figure 17. Results Obtained During Calibration of W-3%Re/W-25%Re Thermocouples Located in the Boiler Section of the Prototype Corrosion Loop. Calibration Test Conducted with a Sodium Flow of 2.5 gpm in the Primary Circuit and no Potassium in the Secondary Circuit.

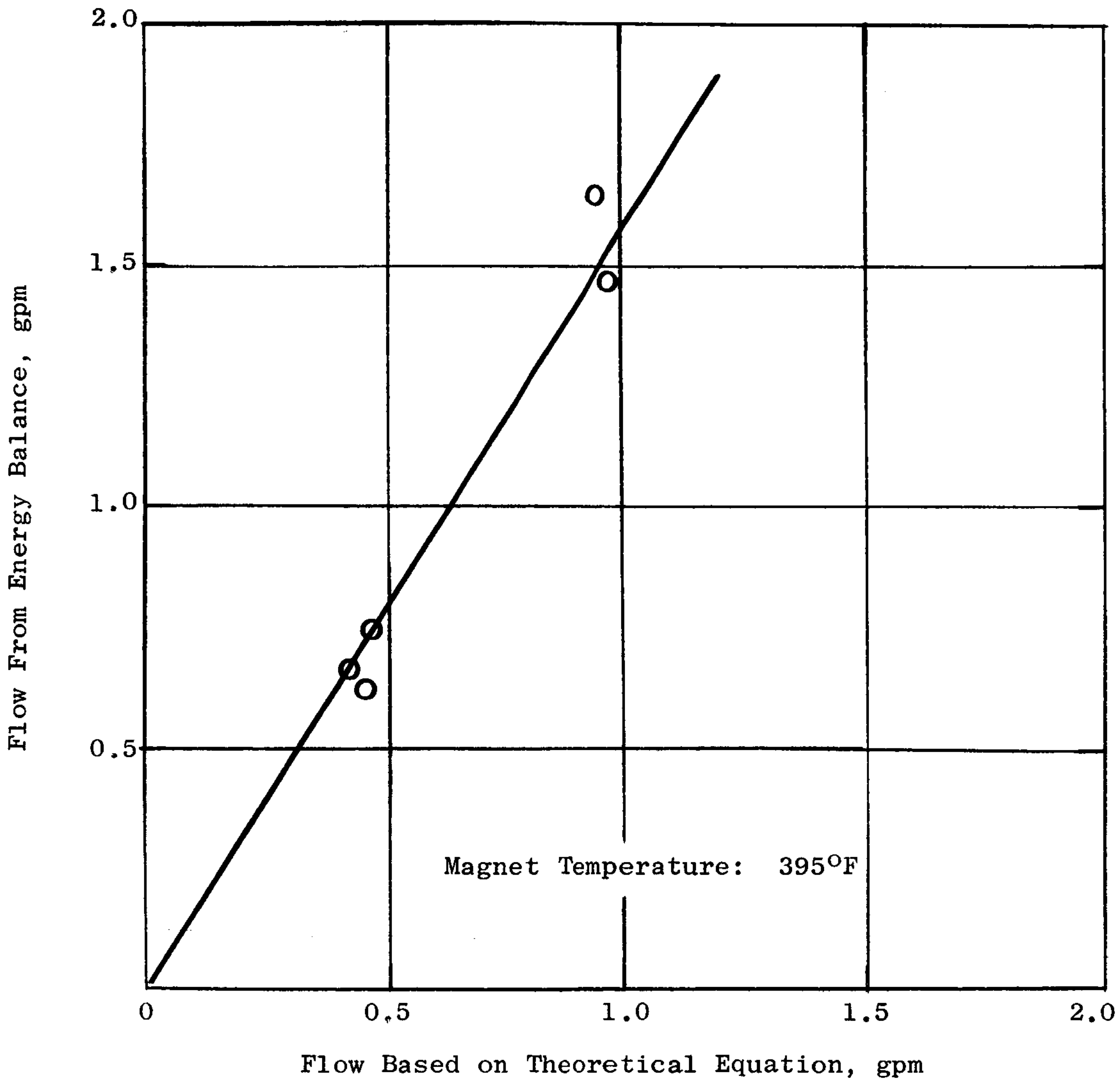


Figure 18. Theoretical Sodium Flow vs Flow Determined by Energy Balance Across Sodium Heater in the Prototype Corrosion Loop.

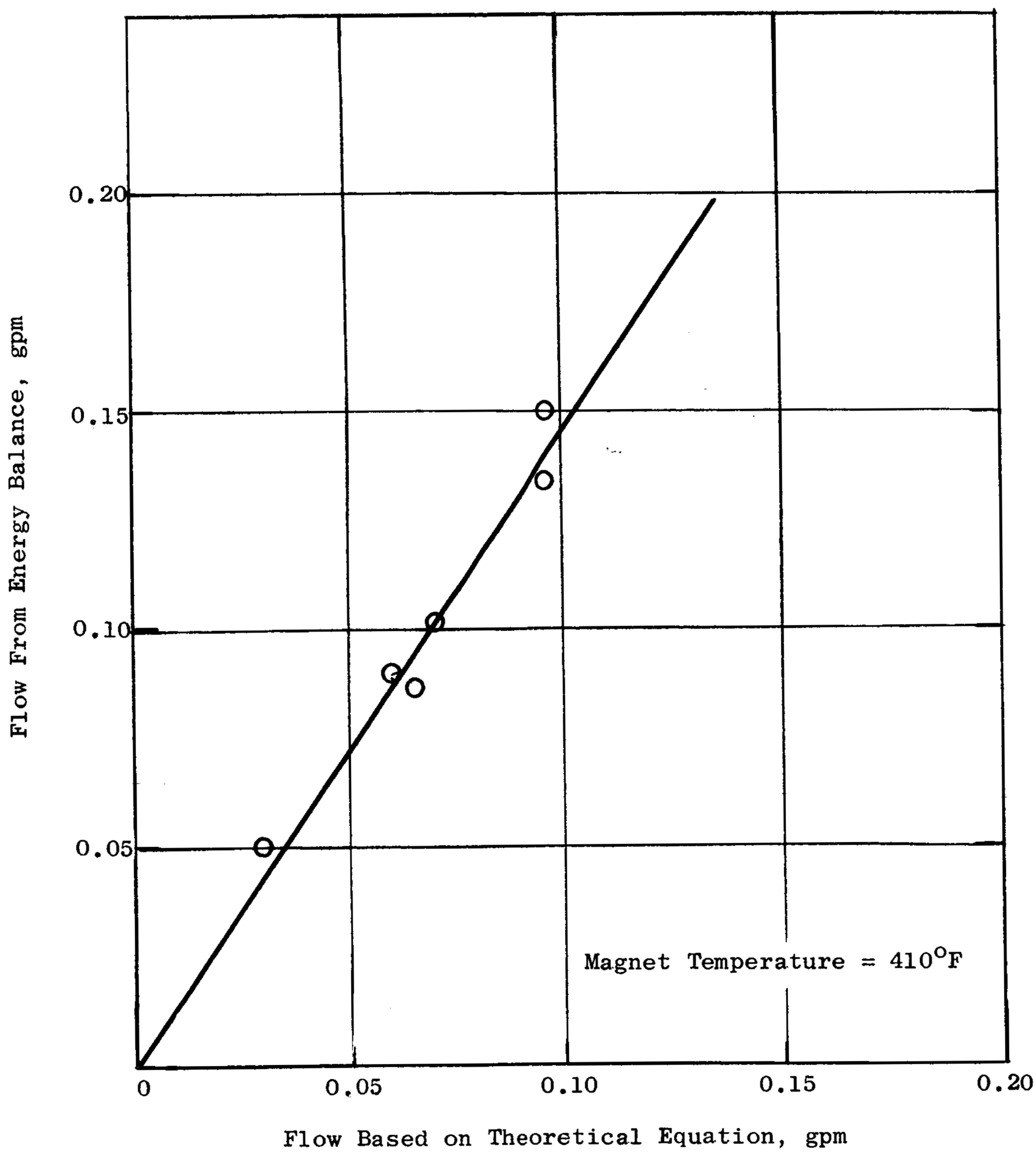


Figure 19. Theoretical Potassium Flow vs Flow Determined by Energy Balance Across Potassium Preheater in the Prototype Corrosion Loop.

and indicated flow rate is not apparent at this time although it may be due to a larger shunting effect of the tube wall on the developed signal than has been assumed in development of the flowmeter equation (7). The Prototype Loop flow tubes are 0.25-inch ID with 0.065-inch walls with a ratio of outer diameter to inner diameter of 1.5:1, which is considerably larger than the diameter ratios previously used (1.12:1) by Affel, et al. in the referenced report (7).

The control console, showing most of the instruments used to monitor loop conditions, is shown in Figure 20. The major instruments not shown are the partial pressure gas analyzer, the getter-ion pump console, the two ionization gauge controls, and the four potassium pressure transducer dial indicators.

After completion of the calibration tests, the loop safety circuits were checked and set according to the test plan. The startup and steady state boiling operation will be reported in the next quarter.

F. Refluxing Potassium Compatibility Tests

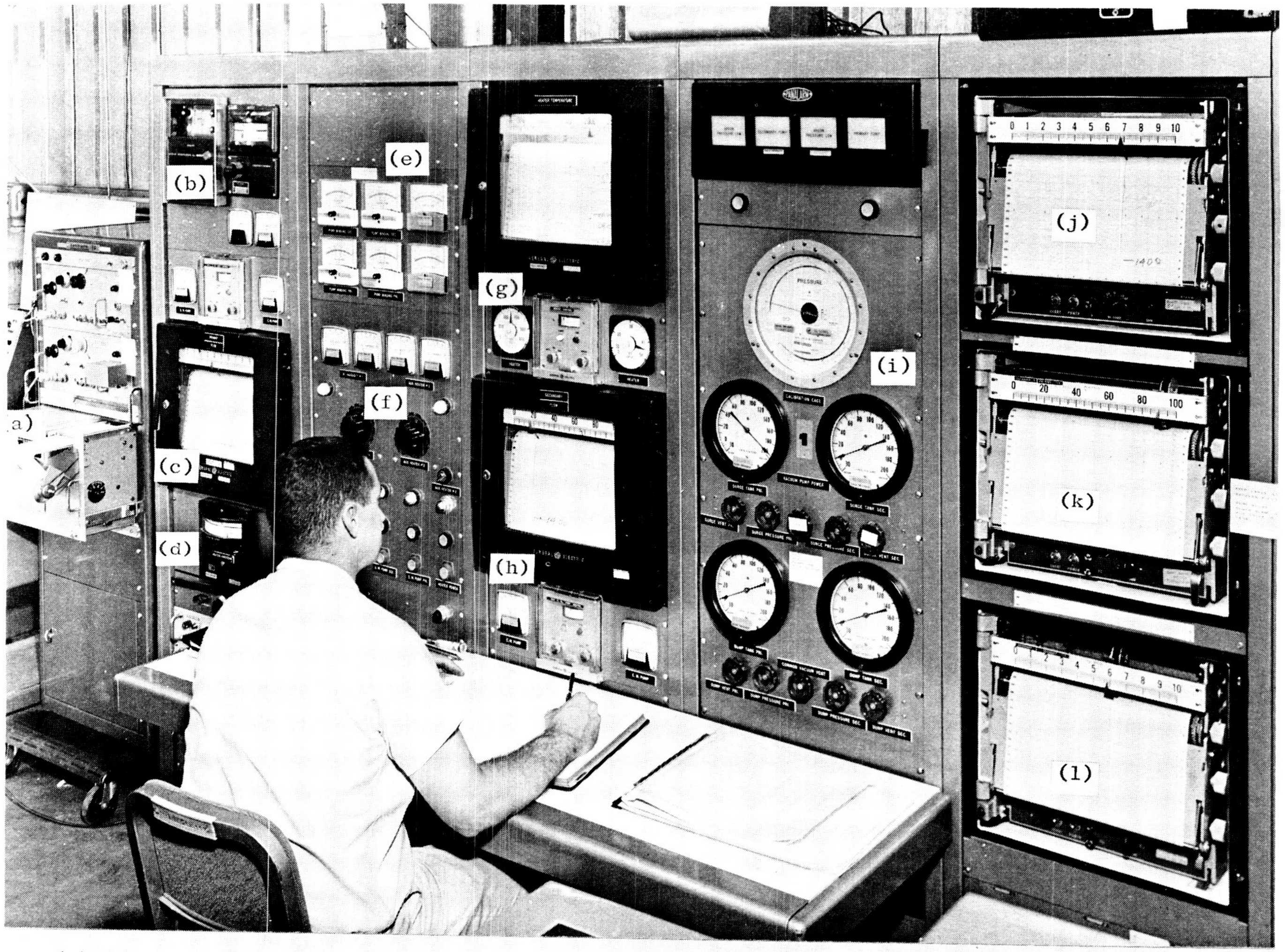
The two refluxing potassium capsule tests which are being conducted at 2000°F to determine the extent of mass transfer of Mo-TZM alloy tubular inserts located in the condenser region of Cb-1Zr alloy capsules have been described in previous reports (9 and 10). The results of the preliminary examination of the test components from the 2500-hour test were reported in a previous report (11). Chemical analyses and metallographic examinations of test components from the two tests have been concentrated on the 5000-hour test which will be described below.

The 5000-hour test was terminated during the past quarter and examination of the test components has been completed. The test chamber pressure during the 5000-hour test is summarized in Table II.

The potassium condensing rate for the first 3300 hours of the 5000-hour capsule test was included in the previous progress report (11). During the 5000-hour test, the condensing rate varied from a minimum value of 27.6 $lbs/ft^2/hr$ to a maximum of 30.9 $lb/ft^2/hr$. The capsule temperature, as determined by thermocouples located in wells in the condenser and boiler region, was $2000^{\circ} \pm 10^{\circ}F$. The condenser thermocouple indicated temperatures 2° to $5^{\circ}F$ less than the boiler thermocouple during the test.

Following completion of the test, the capsule was removed from the test chamber and transferred to the vacuum/purge welding chamber where the capsule was opened in a high purity helium environment. The two parts of capsules were placed in the distillation unit and distilled free of potassium by the method previously described (12).

Following removal from the welding chamber, the capsules halves were inverted and tapped gently to determine if any loose particulate material remained after distillation. None was found. The five Mo-TZM alloy tubular inserts were then removed from the condenser region of the capsule.



(a) 2-Channel Sanborn Recorder
(b) K Preheater Control
(c) Na Pump Control and Flow Recorder
(d) Heater Overtemperature Control
(e) EM Pump Winding Temperatures
(f) Auxiliary Heater Controls
(g) Na Heater Control and Recorder
(h) K Pump Control and Flow Recorder
(i) Vacuum and Argon Pressure Gauges and Manifold
(j) K Pressures and Junction Block Temperatures
(k) Na Circuit Temperatures
(l) K Circuit Temperatures

Figure 20. Control Console of the Prototype Corrosion Loop. (Orig. C65080601)

TABLE II. TEST CHAMBER PRESSURE DURING 2500-HR AND 5000-HR, 2000°F REFLUXING POTASSIUM CAPSULE TESTS

Test Period	Pressure, Torr
Initial pumpdown	2.0×10^{-8}
Chamber at 60°F after 14 hr of bakeout at 400°F	2.2×10^{-9}
Capsules heated to 2000°F over a 45 hr period	1×10^{-6} (max.)
0-20 hr	9×10^{-7} to 2×10^{-7}
20-100 hr	2×10^{-7} to 1×10^{-7}
100-500 hr	1×10^{-7} to 2×10^{-8}
500-2500 hr	2×10^{-8} to 6×10^{-9}
2500 hr	2500-hour capsule removed from test chamber
Chamber at 60°F after 14 hr of bakeout at 400°F	2.3×10^{-9}
5000-hr capsule heated to 2000°F over a 2-1/2 hr period	2.0×10^{-7} (max.)
2500-2540 hr	2×10^{-7} to 7.8×10^{-9}
2540-5000 hr	7.8×10^{-9} to 5×10^{-9}
Chamber at 60°F following completion of test	9×10^{-10}

Weight change data on the five Mo-TZM alloy insert specimens and the results of chemical analysis on the inner 10 mils of the insert specimens are given in Table III. The largest weight loss observed was approximately thirty times that of the largest weight loss observed in the 2500-hour test but was still small enough to be considered insignificant, being equivalent to 0.00005-inch of uniform surface removal. The results of chemical analyses indicate no significant change in the oxygen, nitrogen, or hydrogen concentrations of the specimens. A slight decrease in the carbon concentration was noted and will be discussed later in this section.

The appearance of the various capsule components is illustrated in Figure 21(a) and is quite similar to the appearance of the components from the 2500-hour test (13). The areas of the Cb-1Zr capsule in contact with liquid potassium (boiler region and the region containing the Mo-TZM alloy inserts) were bright, while the vapor region above the liquid-vapor interface had a darkened appearance. As may be noted in Figure 21(a), the portions of the Cb-1Zr sheet specimen which extended above the liquid-vapor interface was also darkened during the test. Spectrographic analysis was performed on scrapings taken from the dark regions and the bright regions of the Cb-1Zr sheet specimens to determine if the discoloration might be due to transfer of Mo or Ti from the Mo-TZM alloy specimens located in the condenser region. No differences were detected. In addition, no variation in the zirconium concentration in the two regions could be detected.

The results of chemical analysis performed on various samples of the Cb-1Zr capsule and the sheet specimen are given in Table IV. No significant changes in the nitrogen, hydrogen and carbon concentrations were observed. The results indicate a buildup of the oxygen level in the boiler (liquid) region and the darkened regions of the capsule and in the sheet specimen above the liquid-vapor interface. The results obtained on the inner 10 mils of the boiler wall and the total cross section and core (inner 60 mils) of the sheet specimen indicate that the bulk of oxygen pickup via the potassium is concentrated near the surface of the Cb-1Zr components. The apparent oxygen migration from the condenser region to the boiler region is consistent with results obtained in refluxing potassium Cb-1Zr capsule tests at General Electric (14) and Oak Ridge National Laboratory (15).

Calculations of the oxygen increase in the Cb-1Zr specimen in the boiler region plus the increase noted in the inner 10 mils of the boiler region wall indicate an oxygen addition of approximately 25 mg during the 5000-hour test. Based on the low oxygen content of the potassium (0.18 mg) and the insignificant change noted in the Mo-TZM alloy specimens, it is tentatively concluded that the principal source of oxygen was from the Cb-1Zr wall in the condenser region, possibly enhanced by the diffusion of oxygen from the external environment through the capsule wall. However, the amount of oxygen contamination from the external environment is very low.

The microstructure of the various capsule components is illustrated in Figure 21. A view of the total cross section of one of the Mo-TZM alloy inserts and enlarged views of the inner and outer surface of this insert are shown in Figure 21(b). No corrosion or surface layers were detected. Varying amounts of recrystallization were detected in the Mo-TZM alloy specimen. The structure of

TABLE III. WEIGHT CHANGE AND CHEMISTRY DATA OBTAINED ON Mo-TZM ALLOY INSERT SPECIMENS FROM THE CONDENSER REGION OF A Cb-1Zr CAPSULE FOLLOWING EXPOSURE TO POTASSIUM FOR 5000 HOURS AT 2000°F[1]

Specimen	Weight, gm		Weight Change		Chemical Analysis, ppm[3]			
	Before	After	mg	mg/cm^2 [2]	O	N	H	C
Mo-TZM[4] #6 (Top of Condenser)	26.6525	26.6358	-16.7	-1.2				
Mo-TZM #7	31.6158	31.5974	-18.4	-1.3	22	4	2	120
Mo-TZM #8	31.6250	31.6250	0	0				
Mo-TZM #9	31.5949	31.6155	+20.6	+1.4	61	7	5	100
Mo-TZM #10 (Bottom of Condenser)	31.6504	31.6519	+1.5	+0.1				
Mo-TZM Before Test Specimen (MCN 103)	--	--	--	--	39	7	5	190

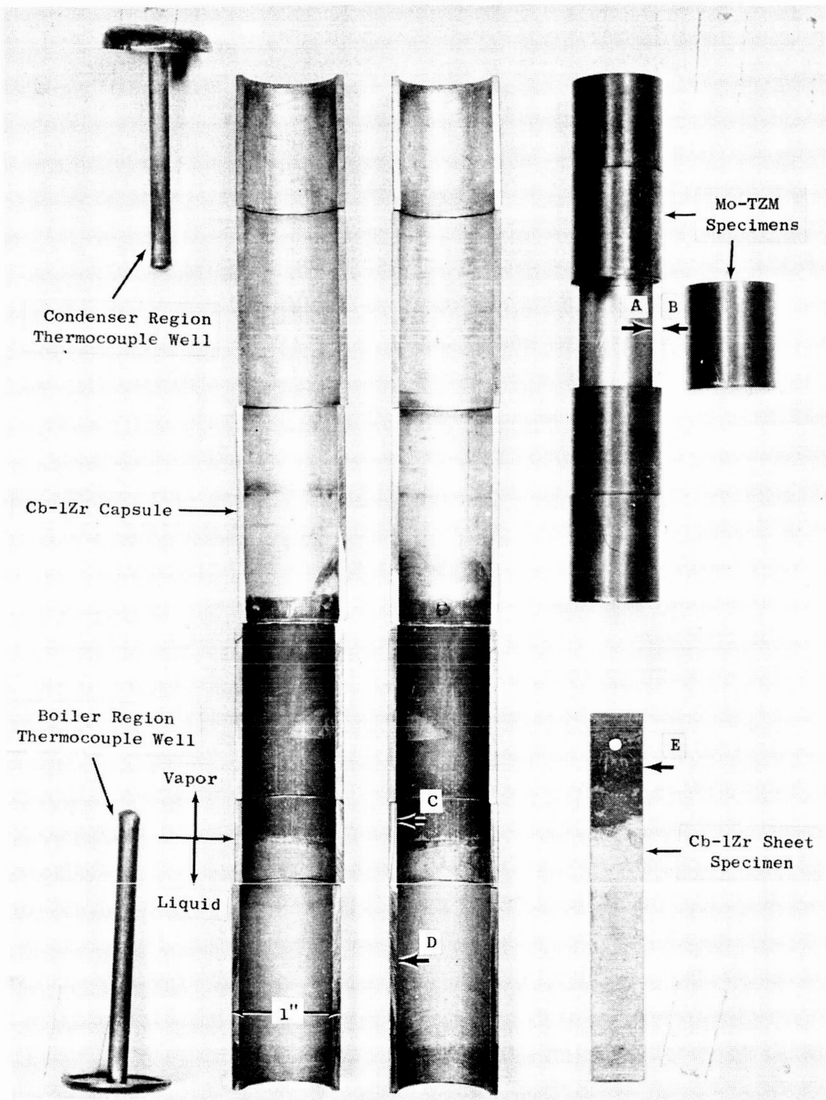
[1] Average potassium condensing rate: 29.0 $lbs/ft^2/hr$ (0.24 $gm/cm^2/min$).

[2] Loss of 25.7 mg/cm^2 for Mo-TZM alloy is equivalent to 1 mil of uniform surface removal.

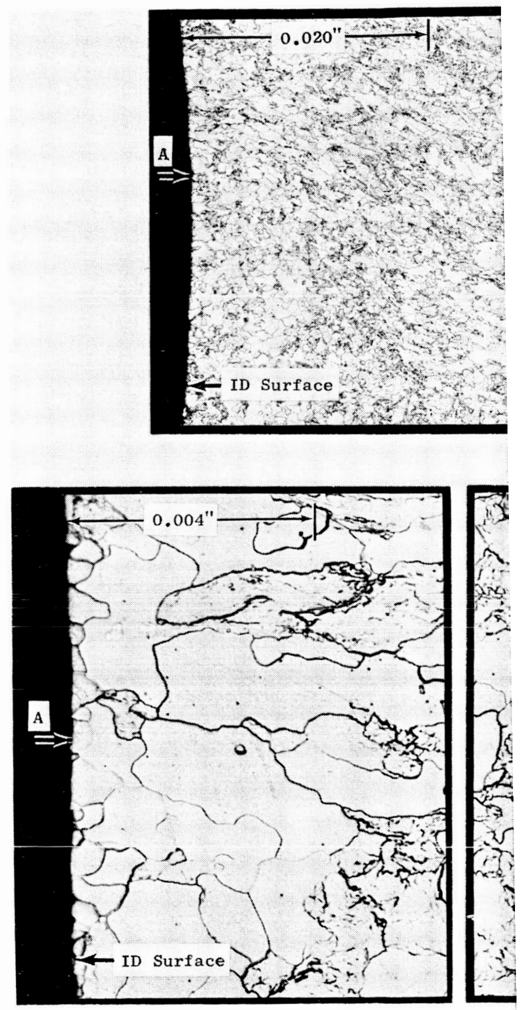
[3] Analyses performed on inner 0.010-inch of wall of tubular inserts. Values given are the average of duplicate analyses.

[4] Nominal dimensions of Mo-TZM alloy insert specimens (inches):

	Length	OD	Wall Thickness
Specimen #6	0.85	0.85	0.078
Specimen #7, #8, #9 and #10	1.0	0.85	0.078

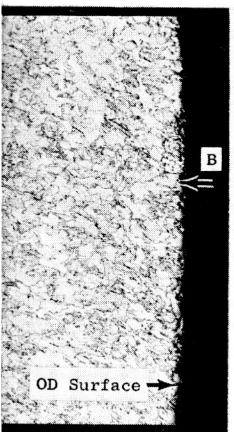


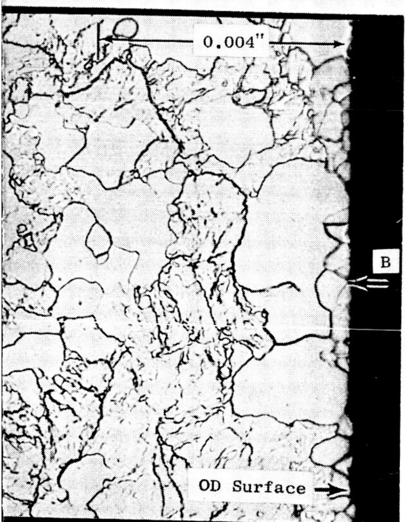
a) Capsule Components (Orig. C65083169)



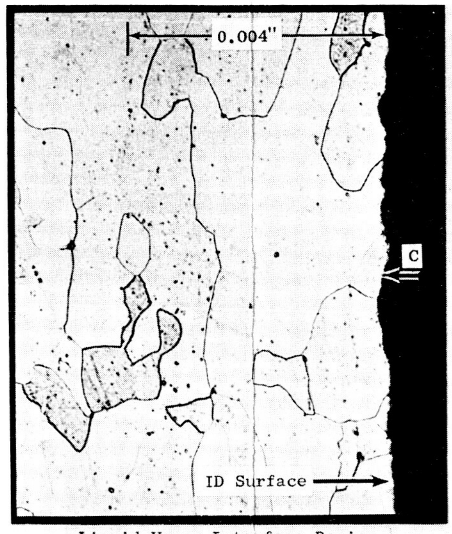
b) Microstructure o

Figure 21. Potassium Reflux Capsule Co
2000°F.

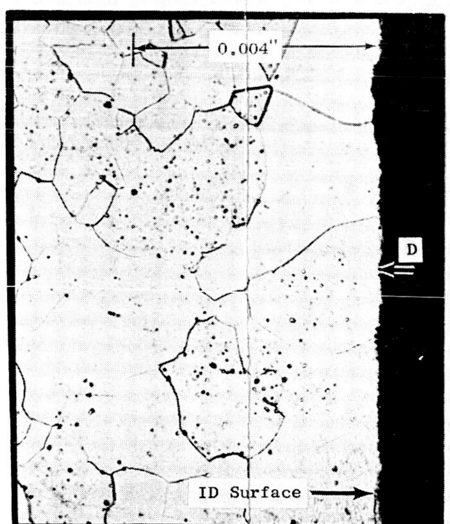




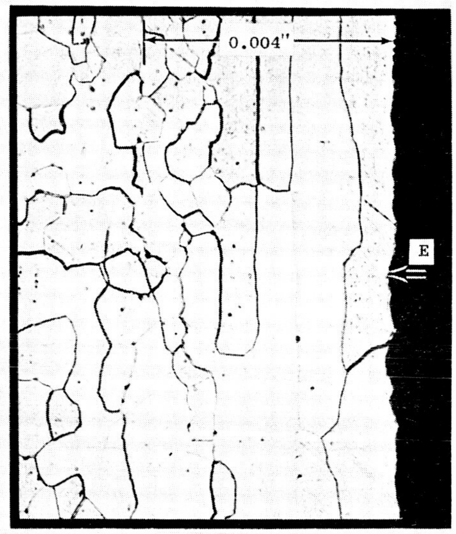
f Mo-TZM Specimen



Liquid-Vapor Interface Region



Boiler Region



Sheet Specimen

c) Microstructure of Cb-1Zr Test Components

ıponents Following 5000-Hour Test at

2

TABLE IV. RESULTS OF CHEMICAL ANALYSIS OF Cb-1Zr CAPSULE FOLLOWING COMPLETION OF 5000-HOUR, 2000°F REFLUXING POTASSIUM TEST[1,2]

	Chemical Analysis, ppm[3]			
	O	N	H	C
Capsule Wall[4,5]				
Condenser Region Wall	268	93	5	100
Inner 0.010-Inch of Condenser Wall	240	104	6	90
Vapor Region 1-Inch Above Liquid-Vapor Interface, Inner 0.010-Inch of Wall	291	106	8	50
Boiler Region Wall	272	96	7	60
Inner 0.010-Inch of Boiler Wall	427	114	9	80
Before Test	184	95	1	40
Sheet Specimen from Boiler Region[6]				
Dark Region Above Liquid-Vapor Interface	533	24	5	70
Bright Region Below Liquid-Vapor Interface	472	25	7	90
Following Removal of 0.010-Inch From Each Surface	289	36	5	-
Before Test	210	33	2	40

[1] Mo-TZM alloy tubular inserts located in the condenser region.

[2] Average potassium condensing rate: 29.0 lbs/ft^2/hr (0.24 gm/cm^2/min).

[3] Values given are the average of duplicate analyses.

[4] Nominal dimensions of Cb-1Zr capsule: 1-inch OD x 0.075-inch wall x 10 inches long.

[5] Analyses are for total wall unless otherwise specified.

[6] Nominal dimensions of Cb-1Zr sheet specimen: 0.5-inch wide x 0.079-inch thick x 3.5-inch long. Specimen weighed 18.9019 gm before test and gained 6.9 mg (0.26 mg/cm^2).

the inner wall of the capsule was approximately 75 percent recrystallized to a depth of 10 mils and the remainder of the wall was approximately 40 percent recrystallized. The predominance of the recrystallized structure near the surface, the slight carbon reduction noted in the inner 10 mils of the Mo-TZM specimen (Table III), and the slight increase in carbon concentration noted in the Cb-1Zr specimens suggests that decarburization of the Mo-TZM alloy may have contributed to the recrystallization gradient noted.

The metallographic appearance of the surface of the Cb-1Zr capsule in the liquid region and the vapor region above the liquid-vapor interface is shown in Figure 21(c). No corrosion was observed in either these specimens or the sheet specimen which is also shown. A special effort was made to retain any possible surface layers which might be responsible for the dark appearance of the vapor region of the Cb-1Zr alloy capsule. The specimens from this region were nickel plated prior to metallographic preparation to aid in edge preservation. A discontinuous layer with a maximum thickness of 0.1 mil was detected. This layer, which was very fragile, is thought to contain a high concentration of oxygen and may be a complex compound(s) of oxygen, potassium, columbium, and zirconium of the type suggested by the studies of Litman (16).

It is concluded from the results of this 5000-hour test and the 2500-hour test which preceded it that Mo-TZM alloy is compatible with condensing potassium under these tests conditions and the results further substantiate the selection of the Mo-TZM alloy as the turbine alloy for evaluation in the Cb-1Zr Prototype Corrosion Loop.

IV FUTURE WORK

A. The Prototype Corrosion Loop will be put in operation at the test design conditions.

B. Preparation of topical reports covering the various portions of the program which have been completed will continue.

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(14) NASA Contract NAS 3-2140.

(15) Metals and Ceramics Division Annual Progress Report for Period Ending June 30, 1965, ORNL-3870, p 128.

(16) Litman, A. P., "The Effect of Oxygen on the Corrosion of Niobium by Liquid Potassium," ORNL-3751, July, 1965.

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